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The Journal
of the
Society of Dyers and Colourists

Volume 73 Number 9

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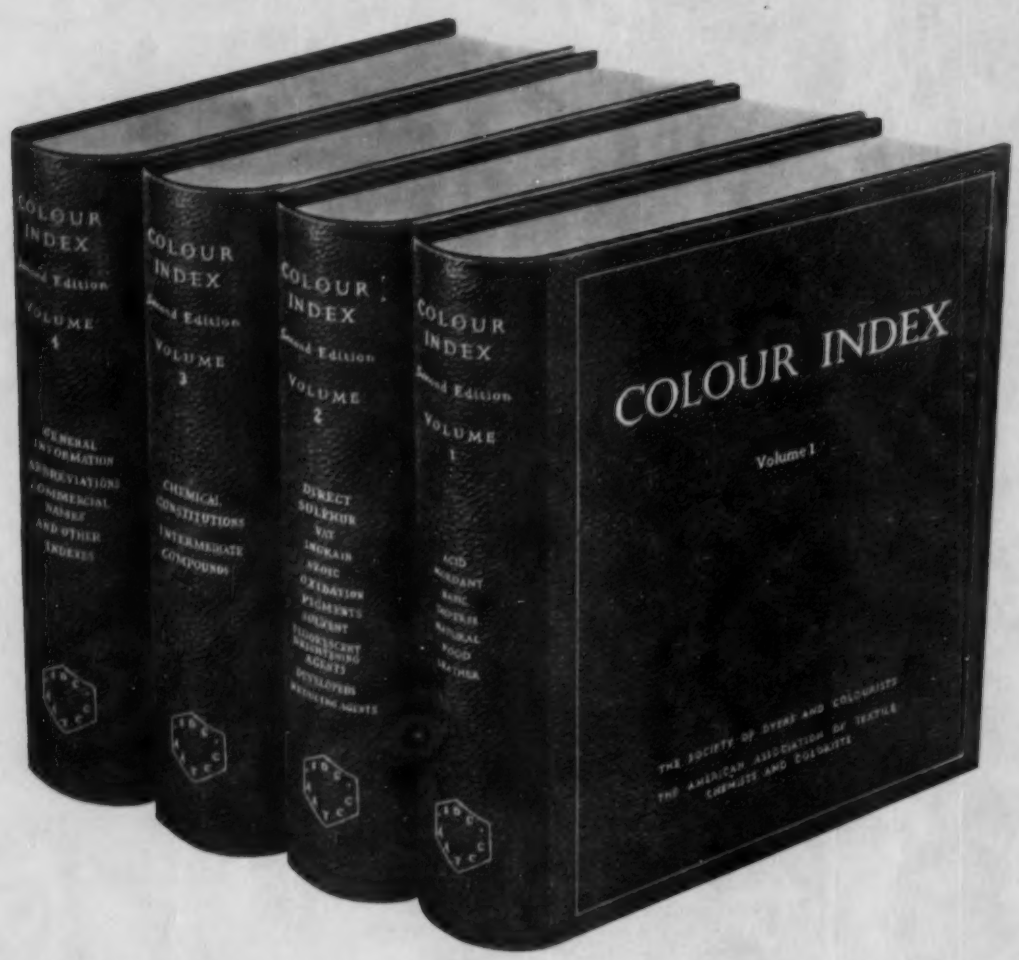

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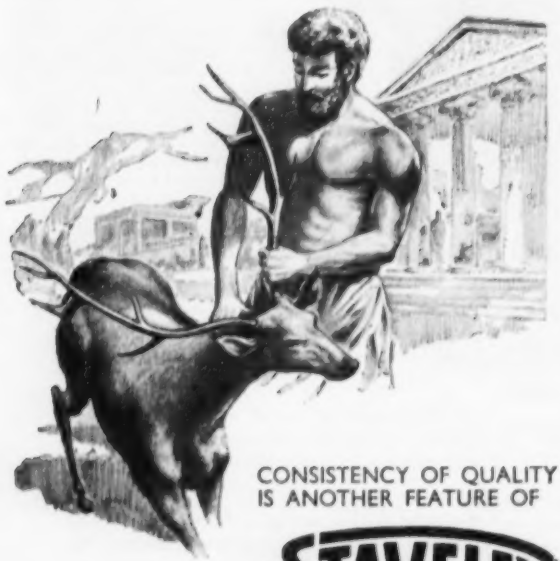

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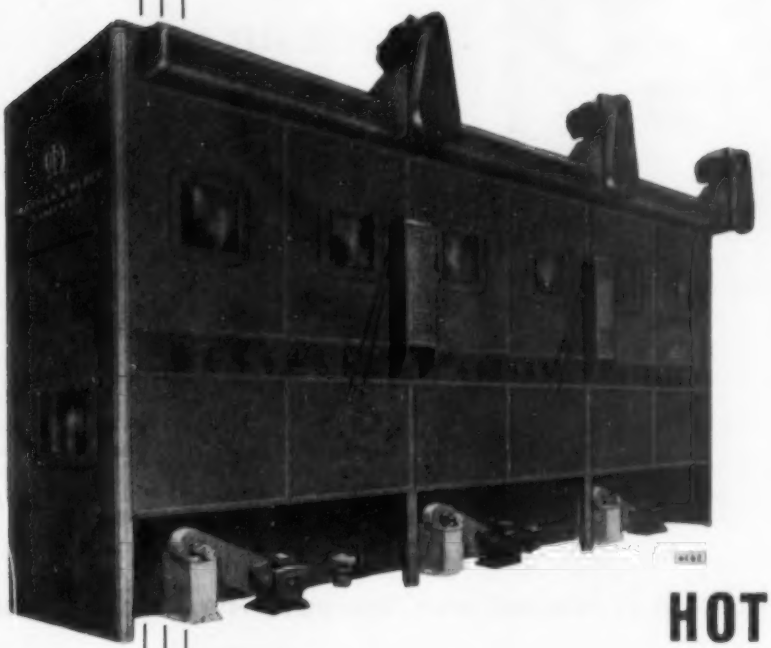

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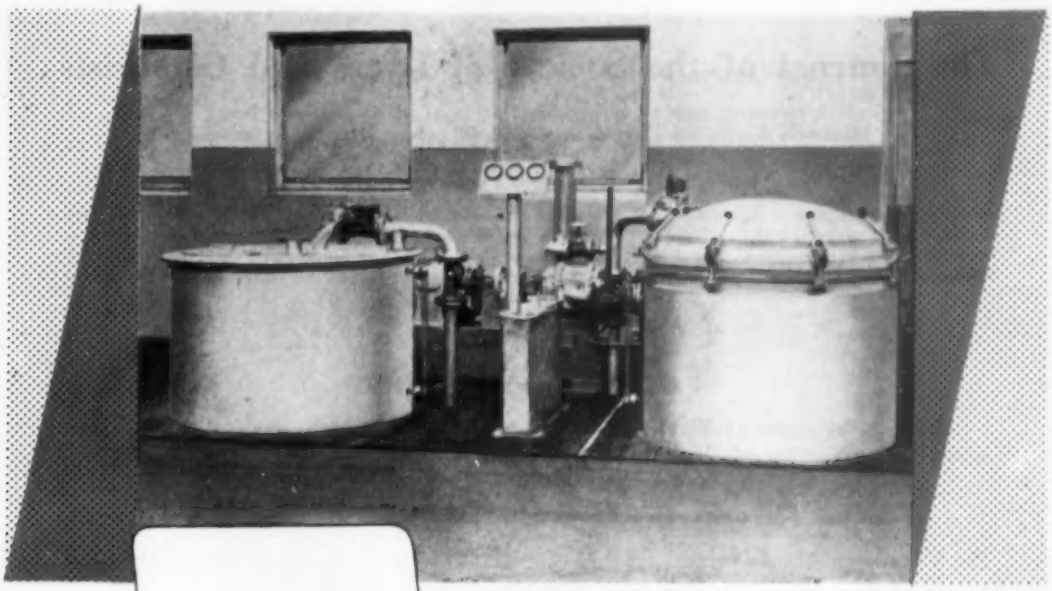

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–7 of the January 1957 and pages 285–290 of the July 1957 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

The Variation of Colour Fastness on Fabrics — *S. Horrdin and Ulf Bülow*

The Preparation and Properties of Regenerated Cellulose containing Vinyl Polymers
III— Moisture Relations — *G. Landells, J. A. Leathley, and C. S. Whewell*

The Effect on Wool of Boiling in Aqueous Solutions
III— Acids and Acidic Salt Solutions — *R. V. Peryman*

Relative Stabilities of Metal Derivatives of
oo'-Dihydroxyazo Dyes — *F. A. Snavely, W. C. Fernelius, and B. E. Douglas*

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FORTHCOMING MEETINGS OF THE SOCIETY

Tuesday, 24th September 1957

NORTHERN IRELAND SECTION. *The Uses of Acrilan in 100% Form as well as in Blends with Other Fibres, and The Dyeing and Finishing of Such Materials.* W. H. Hindle Esq. (Technical Director, Chemstrand Ltd., U.S.A.), Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. *Some Experiments on the Effect of Dye, Fibre and Atmosphere on Light Fastness.* Dr. G. Schmidt (Badische Anilin & Soda Fabrik). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Wednesday, 2nd October 1957

MIDLANDS SECTION. *The Shrink Resistance of Wool.* A. N. Davidson, Esq., B.Sc. College of Technology, **Leicester.** 7 p.m.

Friday, 4th October 1957

LONDON SECTION. *Flame-proofing of Textile Fabrics, with particular reference to the function of Antimony compounds.* N. J. Read, Esq., A.C.G.I., F.R.I.C. (Associated Lead Manufacturers Ltd.). (Joint meeting with the London Branch of the Clothing Institute.) Royal Society, Burlington House, **London, W.1.** 6 p.m.

Friday, 11th October 1957

LONDON SECTION. Fifth London Lecture. *The Art of Costume.* Miss Anne M. Buck, Keeper of the Gallery of English Costume, Manchester. Waldorf Hotel, **London, W.C.2.** 7 p.m.

WEST RIDING SECTION. Supper and Smoker. Victoria Hotel, **Bradford.** 7.30 for 8 p.m.

Tuesday, 15th October 1957

SCOTTISH SECTION. *Dyeing and Finishing of Fibro Blends.* W. Lonie, Esq., B.Sc., Ph.D. (British Rayon Research Association). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

HUDDERSFIELD SECTION. *Recent Aspects of Silicone Water Repellents.* T. W. Watson, Esq. (Midland Silicones Ltd.). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

Wednesday, 16th October 1957

SCOTTISH JUNIOR BRANCH. *Dyeing and Finishing of Fibro Blends.* W. Lonie, Esq., B.Sc., Ph.D. (British Rayon Research Association). Technical College, **Paisley.** 7.30 p.m.

Thursday, 17th October 1957

MANCHESTER JUNIOR BRANCH. *The Scope of the Dyeing Technologist in the Textile Industry.* H. A. Thomas, Esq., B.Sc., Ph.D., F.R.I.C., F.T.I. Manchester College of Technology, **Manchester.** 4.30 p.m.

Friday, 18th October 1957

NORTHERN IRELAND SECTION. Dinner Dance. Woodbourne House Hotel.

Saturday, 19th October 1957

MIDLANDS SECTION. Ladies' Evening. Dinner Dance. Grand Hotel, **Leicester.**

Wednesday, 30th October 1957

MIDLANDS SECTION. *New Developments in the Winch Dyeing of Cotton Knitted Fabrics.* C. Newton, Esq., A.M.C.T. King's Head Hotel, **Loughborough.** 7 p.m.

Thursday, 31st October 1957

WEST RIDING SECTION. *The Dyeing of Acrilan and its Blends.* H. R. Hadfield, Esq., M.Sc.Tech. (Imperial Chemical Industries Ltd.). Victoria Hotel, **Bradford.** 7.30 p.m.

Friday, 1st November 1957

LONDON SECTION. *Problems of Early English Calico Printing.* Peter Floud, Esq., C.B.E., Keeper of Circulation, Victoria and Albert Museum. (Joint meeting with the London Section of the Textile Institute.) Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 12th November 1957

SCOTTISH SECTION. *New Methods for the Assessment of the Dyeing Characteristics of Neutral-dyeing Wool Dyes.* Dr. H. R. Hirsbrunner (J. R. Geigy S.A., Basle). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

This paper will contain many interesting data on the dyeing characteristics of the metal-complex dyes compared with those of the Polar type, and in addition will present a new interpretation of the exhaustion properties of these dyes under varying conditions.

Tuesday, 12th November 1957

NORTHERN IRELAND SECTION. *The Dyeing and Finishing of Fibro Blends* W. Lonie, Esq., B.Sc., Ph.D. (British Rayon Research Association). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Wednesday, 13th November 1957

SCOTTISH JUNIOR BRANCH. *Synthetic Detergents in the Textile Industry.* H. Carter, Esq., A.R.I.C. (Shell Chemicals Ltd.). Technical College, **Paisley.** 7.30 p.m.

Thursday, 14th November 1957

MANCHESTER JUNIOR BRANCH. *Bleaching.* (The Bleachers Association.) Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. *New Methods for the Assessment of the Dyeing Characteristics of Neutral Dyeing Wool Dyes.* Dr. H. R. Hirsbrunner (J. R.Geigy S.A., Basle). Victoria Hotel, **Bradford.** 7.30 p.m.

Friday, 15th November 1957

HUDDERSFIELD SECTION. Annual Dinner.

Tuesday, 19th November 1957

HUDDERSFIELD SECTION. *New Methods for the Assessment of the Dyeing Characteristics of Neutral Dyeing Wool Dyes.* Dr. H. R. Hirsbrunner (J. R. Geigy S.A., Basle, Switzerland). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

MIDLANDS SECTION. *Flameproofing.* T. H. Morton, Esq., M.Sc., Ph.D., F.S.D.C. and F. Ward, Esq. Gas Board Theatre, **Nottingham.** 7 p.m.

SCOTTISH SECTION. Sectional Dance. Oxford Suite of Burlington House, Bath Street, **Glasgow.** 7.30 p.m. to 12 midnight.

Tuesday, 26th November 1957

NORTHERN IRELAND SECTION. *Terylene Polyester Fibre in the Linen Industry.* P. W. Eggleston, Esq., B.Sc., A.T.I. (Imperial Chemical Industries Ltd.). (Joint Meeting with the Textile Institute.) Kensington Hotel, **Belfast.** 7.30 p.m.

Thursday, 28th November 1957

WEST RIDING SECTION. *Problems in the Dyeing of Fast to Washing Colours on Wool Yarn.* J. F. Gaunt, Esq., Ph.D., F.R.I.C., F.T.I. (Patons & Baldwins Ltd.). Metropole Hotel, King Street, **Leeds.** 7.30 p.m.

Tuesday, 3rd December 1957

SCOTTISH JUNIOR BRANCH. *The Measurement of Damage to Wool Fibres.* J. C. Brown, Esq. (Clayton Dyestuffs Co. Ltd.). Technical College, **Paisley.** 7.30 p.m.

Friday, 6th December 1957

LONDON SECTION. *Colour in Writing and Copying.* J. P. Gill, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 10th December 1957

NORTHERN IRELAND SECTION. *An Impartial Observer's View of the Bleaching and Dyeing Industry— A Consultant Speaks Out.* T. L. Collyer, Esq., C.B.E., A.M.I.Mech.E., A.M.I.I.A. Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. *Dyeing of Acrilan.* H. R. Hadfield, Esq., M.Sc.Tech. (Imperial Chemical Industries Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Recent developments in the dyeing of Acrilan will be reviewed and the dyeing and fastness properties of acid, chrome and disperse dyes will be detailed. The dyeing of Acrilan blends will be briefly reviewed.

Wednesday, 11th December 1957

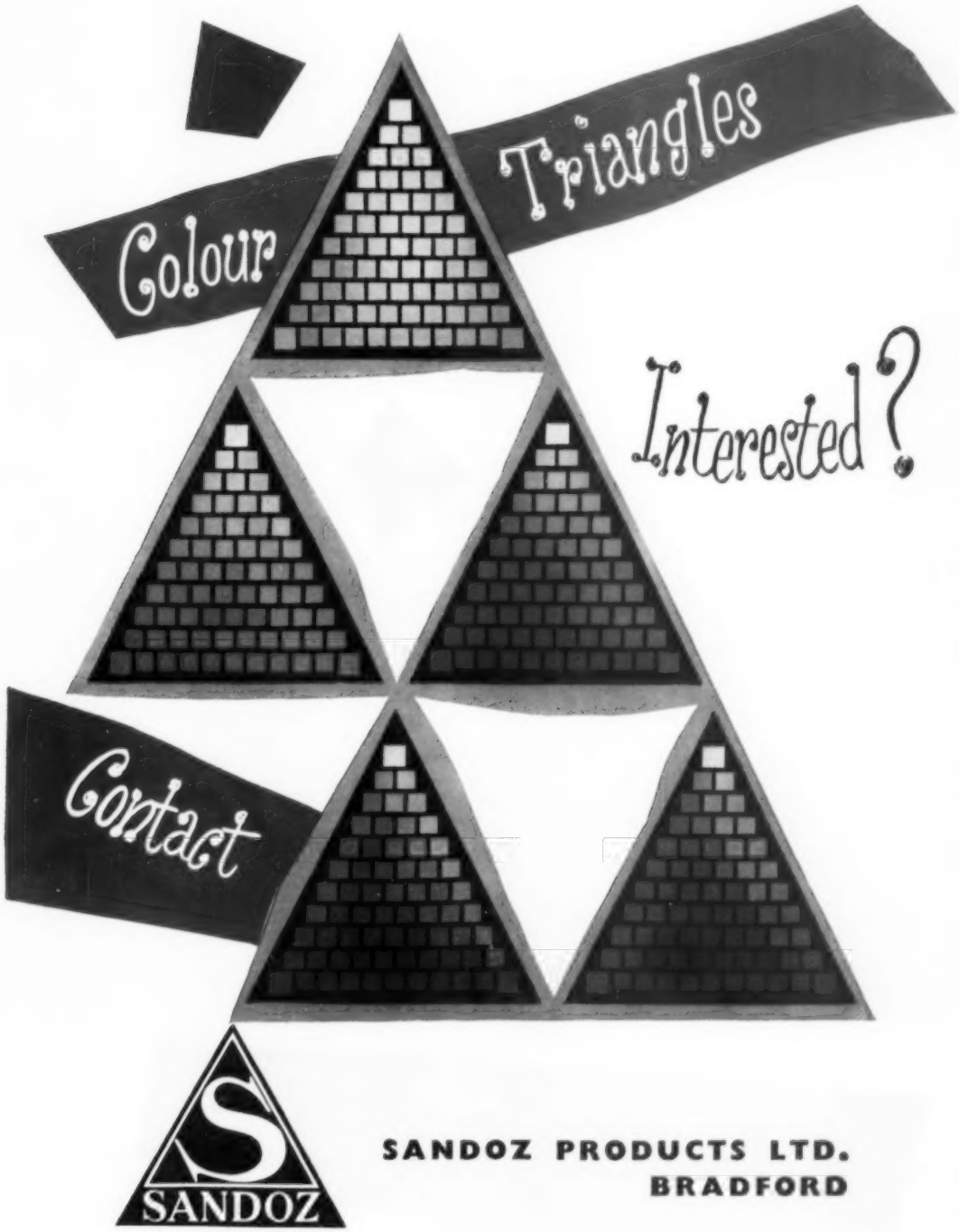
MIDLANDS SECTION. *The Application of Amino Resin Finishes to Cotton.* A. R. Smith, Esq., B.Sc. King's Head Hotel, **Loughborough.** 7 p.m.

Thursday, 12th December 1957

MANCHESTER JUNIOR BRANCH. *The Textile Finisher.* F. Wood, Esq. Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. *Watering of Acetate Poults.* E. France, Esq., B.Sc., A.R.C.S., A.M.C.T. (Courtaulds Ltd., Bocking). Victoria Hotel, **Bradford.** 7.30 p.m.

continued on page xxxvii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THE JOURNAL

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Volume 73 Number 9 SEPTEMBER 1957 *Issued Monthly*

COMMUNICATIONS

The Use of Paper Chromatography in the Recognition of Chemical Damage to Wool

VIBEKE KÖPKE and BAILLI NILSSEN

Hydrolysis of wool has been carried out by means of acid, alkali, and papain, the resulting solutions being analysed by one- and two-dimensional chromatography. Acid hydrolysis appears to be the most convenient method. Experiments on desalting the amino-acid solutions by the method of Consden, Gordon, and Martin show that this cannot usually be employed for purifying the solutions, as cysteic acid, methionine, and probably tyrosinesulphonic acid are destroyed during the process. The method has been used for wool treated with alkali, hydrogen peroxide, and sulphuric acid. In the present form the method is sensitive only to severe damage.

Introduction

Though a number of methods have been proposed for the investigation of chemical damage to wool, hardly any of them has a direct connection with specific chemical reactions of the degrading agent, and therefore they often leave doubts as to the type of damage. As some of the more usual reagents causing damage, viz. alkalis, oxidising agents, and sulphuric acid, should produce definite alterations in some of the amino acids of wool keratin, it was thought worthwhile investigating the possibilities of using paper chromatography to recognise such damage.

The reactions particularly considered were the formation of lanthionine by alkali treatment, of cysteic acid by oxidising agents, and of tyrosinesulphonic acid by sulphuric acid.

Experimental

MATERIAL

All experiments were carried out on Australian 66/70s wool extracted with petroleum ether and alcohol, and then rinsed in water. The wool was opened, and mechanically retained impurities were removed by means of tweezers.

HYDROLYSIS

The wool was hydrolysed in three different ways—

(*a*) By boiling with 6 N. hydrochloric acid, by the method of Block [1]

(*b*) By boiling 20 mg. of wool with a solution of 11 g. of barium hydroxide $Ba(OH)_2,8H_2O$ in 11 ml. of water for 9 hr.

(*c*) By means of papain, according to the method of Lennox [2]. Different preparations of papain were tested: one kindly put at our disposal by Dr. E. G. Lennox and one from E. Merck (Darmstadt, Germany) gave identical results.

DESALTING

Inorganic acids and salts which interfere with the location and separation of the amino acids were in some cases removed by desalting in 10% *iso*propanol solution. The principle of the method is described by Consden, Gordon, and Martin [3], and the apparatus used (with a circulating mercury cathode and a platinum anode) was constructed by the Shandon Scientific Co., London S.W.7.

CHROMATOGRAPHY

In the two-dimensional chromatography of the amino acid solutions the ascending method of Dent [4] was used. The paper used was, however, Schleicher & Schüll No. 2043 B, which gives both better separation and clearer spots. Some change was made also in the dimensions of the sheets and in the solvent combinations. By using sheets 25 cm. × 28 cm. it is possible to obtain the same separation as Dent does by placing paper strips horizontally on top of the chromatograms during the collidine–lutidine run.

The first solvent, phenol–water, was unaltered, whereas the collidine–lutidine–water in the second stage was replaced by ethyl methyl ketone–propionic acid–water (75 : 25 : 30), which gives an equally good separation. It moves, however, more quickly and is less unpleasant to handle. For the separation of the leucine group the method of Work [5] was employed, and for the specific recognition of lanthanionine one-dimensional chromatography was used with *sec.*-butanol–

formic acid–water [6] as solvent. All chromatograms were dried overnight, and developed by steeping in a 0·2% solution of ninhydrin in anhydrous acetone. For the development of proline, a solution of 0·2% isatin in *n*-butanol with 4% glacial acetic acid was sprayed on the chromatograms.

ALKALI TREATMENT

The wool was treated with solutions containing various concentrations of sodium hydroxide and sodium carbonate at different temperatures, as shown in Tables I and II. A constant time of treatment was used, during which the wool was kept in slow motion. The treatment finished, the wool was removed, thoroughly rinsed with water, steeped for a short time in dilute hydrochloric acid, and again rinsed. After drying at room temperature, hydrolysis was carried out with hydrochloric acid, and the amino-acid solutions obtained were desalted. Chromatography was carried out one-dimensionally by the method of Zahn [6] and two-dimensionally as described above.

To produce an alkaline surface attack on the wool, it was treated—(*a*) with 2% alcoholic caustic potash for 2, 8, and 30 min. at room temperature; or (*b*) with 2% aqueous cetyltrimethylammonium hydroxide for 1, 10, and 30 min. at 30°*c*. Aftertreatment and chromatography were as described previously.

HYDROGEN PEROXIDE TREATMENT

In all cases the treatment was made much more severe than in normal industrial bleaching with hydrogen peroxide, because cysteic acid was difficult to recognise in the exploratory experiments. In these the amino-acid solutions produced by hydrolysis with hydrochloric acid were desalted, and the latter process gave rise to the difficulties. By keeping the hydrolysates over soda–lime *in vacuo* for a week before making the final solutions, the cysteic acid could readily be detected in the chromatograms; but even so, a drastic treatment with hydrogen peroxide appeared necessary for its appearance.

The specifications of the hydrogen peroxide treatments are given in Table III.

After treatment, followed by a thorough rinsing in water and drying at room temperature, hydrochloric acid hydrolysis was employed, and both one- and two-dimensional chromatograms were prepared.

SULPHURIC ACID TREATMENT

In the first series of experiments, the wool was boiled in 0·05 N. sulphuric acid (pH = 1·7) for 6 or 9 hr. In the second series the wool was steeped in the sulphuric acid for 30 min., filtered off, and steeped for a short time in fresh acid. The uptake of liquid varied between 23% and 58% based on air-dry wool. Samples of the wool treated as described (second series) were heated for 1 hr. with the temperature rising from 50° to 100°c.; other samples were heated for 2¼ hr. at 90° or 105°c.

In a third series dilute sulphuric acid (1 : 10) was used. In this the wool was steeped for 30 min. or 3 hr., filtered off, and steeped in fresh acid for 5 min. The liquor was removed by suction until 40% of liquor (on air-dry weight) was retained in the sample.

All samples were rinsed in cold water, neutralised with very dilute caustic soda, thoroughly rinsed, and dried at room temperature.

Samples of untreated and treated wool were hydrolysed with—(1) hydrochloric acid, (2) barium hydroxide solution, (3) sulphuric acid, (4) papain. In all cases a desalting was carried out, but even the more drastic treatments failed to give any specific features on the chromatograms which could be interpreted as due to reactions between the sulphuric acid and the amino acids. By hydrolysing with hydrochloric acid and omitting the desalting process, two small spots appeared in the same place on the chromatograms as cysteic acid. For comparison tyrosine was treated with sulphuric acid as described by Städeler [7].

Results and Discussion

METHODS OF HYDROLYSIS AND CHROMATOGRAPHY

(*a*) *Hydrolysis with Hydrochloric Acid*

This method is simple, but to remove all the hydrochloric acid after evaporation the residue must be kept over soda–lime *in vacuo* for approximately a week. Small quantities of residual acid do not disturb one-dimensional chromatograms, but the two-dimensional ones are unsatisfactory when acid remains in the solution. The last traces of acid may be removed by electrolytic desalting. This treatment, however, destroys cysteic acid, methionine, and probably tyrosinesulphonic acid, and thus the use of desalting is excluded for investigating oxidation and sulphuric acid damage. Desalting might be employed for alkali-treated wool, as lanthionine appears to be unaffected by the process.

(*b*) *Hydrolysis with Alkali*

Alkaline hydrolysis appears to attack the amino acids to a considerably greater extent than does acid hydrolysis. To obtain satisfactory chromatograms, a larger amount of hydrolysate is necessary. Cystine and threonine are destroyed, and their reaction products cannot be detected on the chromatograms. According to Bremner [8], however, threonine is converted to glycine and α-aminobutyric acid, cystine to alanine, and some of the serine to glycine and alanine. Apart from α-aminobutyric acid, all these are already present in the normal hydrolysates. Arginine is hydrolysed to ornithine, which is detected in the two-dimensional chromatograms on development with ninhydrin. A spot is also found in the place of methionine sulphone.

(*c*) *Hydrolysis with Papain*

Digestion with papain was never quite complete, the residue amounting with untreated wool to 2–5%. After filtration the hydrolysates could be chromatographed directly.

On the chromatograms a spot appears on the place of methionine sulphoxide, whereas cystine and proline are absent. The absence of cystine is to be expected as a result of the action of the sodium bisulphite, but the explanation of the absence of proline, which is somewhat at variance

TABLE I

Wool treated with Sodium Hydroxide Solutions for 30 min.

Temperature ...	34–38°c.	64–74°c.	90–95°c.
		pH = 10·9	
Fibres	No visible damage	No visible damage	No visible damage
Cystine	Streak	Spot	Spot
Lanthionine ...	None	None	Faint
		pH = 12·0	
Fibres	Faintly yellow, slightly brittle	Heavily damaged	—
Cystine	Spot	Faint streak	—
Lanthionine ...	Moderate	Strong	—

with the findings of Lennox [2, 9], is not so obvious. Proline added to the digestion mixture could be readily detected, and it thus appears that the enzymatic action fails—at least to some extent—when the hydrolysis reaches a proline residue.

The residue after the digestion was carefully washed with water and separately hydrolysed with acid. In the hydrolysate both cystine and proline were found, but the amount of cystine turned out to be very small compared with that found in the normal acid hydrolysis of wool.

Both the velocity and the extent of the digestion with papain vary greatly with the kind of damage to the wool [10]. After excessive alkali treatment the wool retained its fibre structure on normal digestion. The hydrolysate contained in this case all the usual amino acids, but lanthionine could not be detected. This observation is in agreement with the view that breakdown of the disulphide bonds by the reducing agent present is required for the hydrolytic action of the enzyme. The sulphide bonds of lanthionine would not be susceptible to such a breakdown.

In corresponding experiments on wool treated with sulphuric acid, the digestion was more rapid than with normal wool.

All one-dimensional chromatograms using *sec.*-butanol as solvent were very diffuse, and their interpretation is uncertain.

To summarise: alkaline hydrolysis destroys too much of the amino acids to be of practical value. Digestion with papain offers interesting possibilities on the basis of the velocity of hydrolysis, but does not give any special advantages for the final chromatography. Hydrolysis with acid thus appeared to be the best basis for further investigation.

ALKALI-TREATED WOOL

By treatment with weak alkaline solutions some 50% of the cystine in wool may be converted to lanthionine [11]. Both on the one- and the two-dimensional chromatograms, the gradual increase in lanthionine with increasing temperatures and concentrations in the alkali treatment could be followed. The observations are summarised in Tables I and II. An alteration in the appearance of the cystine spot may be noteworthy; whereas untreated wool gave a typical streak in the direction of the phenol, this disappeared when the wool had been treated with alkali, in some cases even before the lanthionine spot could be detected. With samples treated with alkalis known to produce mainly a superficial attack, viz. alcoholic potash and aqueous cetyltrimethylammonium hydroxide, lanthionine could barely be recognised, in the former case not at all, in the latter only as a shadow after more severe treatment. The importance of this observation lies in the fact that such treatments produce a pronounced effect both on the felting properties and on the rate of dyeing of the wool. This possibility appears to have been completely overlooked by Houff and Beaumont [12].

TABLE II

Wool treated with Sodium Carbonate Solutions for 30 min.

Temperature ...	40°c.	50°c.	60°c.
		5 g. Na_2CO_3 PER LITRE	
Fibres	—	No visible damage	No visible damage
Cystine	—	Streak	Spot
Lanthionine ...	—	None	Doubtful
		10 g. Na_2CO_3 PER LITRE	
Fibres	—	No visible damage	No discoloration, slightly brittle
Cystine	—	Faint streak	Spot
Lanthionine ...	—	Doubtful	Very faint
		15 g. Na_2CO_3 PER LITRE	
Fibres	No visible damage	No visible damage	Faintly yellow, brittle
Cystine	Streak	Spot	Spot
Lanthionine ...	None	Very faint	Faint
		20 g. Na_2CO_3 PER LITRE	
Fibres	No visible damage	No discoloration, slightly brittle	Faintly yellow, brittle
Cystine	Streak	Faint spot	Spot
Lanthionine ...	None	Faint	Moderate

TABLE III

Wool (root ends) treated with Hydrogen Peroxide

Concentration ...	9–10% H_2O_2	17–20% H_2O_2	35–40% H_2O_2
	ROOM TEMPERATURE, 45 MIN.		
Fibres	No visible damage	No visible damage	No visible damage
Cystine	Streak	Streak	Spot
Cysteic acid ...	Just recognisable	Faint	Moderate
	50°C., 15 MIN.		
Fibres	No visible damage	No visible damage	Faintly reddish
Cystine	Streak	Streak	Spot
Cysteic acid ...	Moderate	Moderate	Strong
	ON BOILING WATERBATH, 15 MIN.		
Fibres	Faintly reddish, brittle	Reddish yellow, brittle	Fibres disintegrated
Cystine	Spot	Spot	Faint spot
Cysteic acid ...	Strong	Strong	Very strong

WOOL TREATED WITH HYDROGEN PEROXIDE

As already mentioned, the desalting process had to be omitted in order to recognise the cysteic acid. In the clear two-dimensional chromatograms, the gradual increase in cysteic acid content could be observed. In this case, too, the streak formed by cystine disappeared, but only with severely treated wool. Cystine spots remained, however, even when the treatment produced a disintegration of the wool.

The lower limit for a recognisable attack by hydrogen peroxide appears to be treatment with an approx. 10% solution at room temperature for 45 min. One-dimensional chromatography [6] was unsuccessful, as cysteic acid and arginine are not separated even on Schleicher & Schüll paper No. 2043 B.

ACID-TREATED WOOL

It is known that cystine is not readily attacked by treatment with concentrated sulphuric acid [13, 14]. Reitz *et al.*[13] have shown that it is principally serine which reacts with sulphuric acid, forming a sulphuric ester. They have further shown that tyrosine reacts and gives a sulphonic acid in amounts depending on the conditions of treatment. On hydrolysis with acid or alkali the sulphuric esters are decomposed, whereas the sulphonic acid remains intact. A search for tyrosinesulphonic acid therefore seemed appropriate in the case of wool treated with sulphuric acid.

As mentioned above, hydrolysis of such wool with hydrochloric acid without electrolytic desalting gave rise to two small spots approximately in the same position as the cysteic acid spot. Furthermore, tyrosine treated with sulphuric acid gave a spot in the same place. It is thus highly probable that one of the spots found in the case of wool treated with sulphuric acid is due to tyrosinesulphonic acid.

Conclusions

With the methods investigated, only severe alkali damage on wool can be detected. The important surface changes brought about by limited attack escape recognition. Oxidation damage also must apparently be very serious to be recognisable.

In some special cases, particularly in connection with carbonisation faults, chromatography may be a useful method, but owing to the proximity of the cysteic acid and tyrosinesulphonic acid spots, a sure distinction between the types of damage will apparently necessitate investigations with other solvent combinations for the two-dimensional chromatography.

* * *

The authors are indebted to the International Wool Secretariat for financial aid which enabled this investigation to be carried out.

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(*Received 28th February 1957*)

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Reclamation of Fibres from Rags
I—Identification of Non-carbonisable Fibres

J. C. ATKINSON and J. B. SPEAKMAN

Staining tests to distinguish between carbonisable, non-carbonisable, and protein fibres, and between the chief non-carbonisable fibres, have been developed. The fibres are treated with a mixture of fluorescent compounds, in presence of a stripping agent if dyed, and are then examined in ultraviolet radiation. In a few cases, notably when the non-carbonisable fibres are heavily dyed black, stripping should precede staining.

In the past, when the fibres most likely to be mixed with wool were cotton and viscose rayon staple fibre, the recovery of wool for re-use presented little difficulty, because the cellulosic fibres are readily attacked by acids and can be removed, with little damage to the wool, by treatment with hot gaseous hydrogen chloride. Some difficulty was encountered with fabrics containing silk, and in so far as the latter was replaced by rayon, it might be claimed that the advent of man-made fibres was at first beneficial to the low-woollen industry. But then came the development of cellulose acetate, nylon, Terylene, and Orlon, all of which resist destruction by acids, and the growing use of blends of wool and staple fibres of these kinds is a cause of serious concern in the low-woollen industry. There is at present no satisfactory commercial method of recovering wool from such blends for re-use, either by destroying the second component or, better, since the newer man-made fibres are of similar value to wool, by separating the components of the blend without destroying either.

It seems unlikely that any single process will be found capable of separating the component fibres of all blends, and a rapid method of identifying the non-carbonisable fibres present is clearly needed in order to facilitate classification for subsequent treatment. One possibility, which is the subject of this paper, is to treat the materials with a solution containing several fluorescent compounds, each having an affinity for some particular man-made fibre, and then to identify the latter by the colour of the fluorescence in ultraviolet radiation. Dyes were chosen as the fluorescent compounds to give additional ease of identification of undyed fibres, but it was hoped that dyed materials might be classified, after staining but without stripping, by examination in ultraviolet radiation.

Experimental

UNDYED FIBRES

In relation to the processes of the low-woollen industry, the fibres present in rags may be regarded as falling into three groups, viz. cellulosic fibres, which are carbonisable, non-carbonisable man-made fibres, and protein fibres. At first, therefore, attention was focussed on the preparation of a mixture of dyes which would distinguish sharply between the three groups, the assumption being that any protein fibres present would normally be wool or a related animal fibre. Failure to discriminate between wool and fibres such as Ardil and Fibrolane was regarded as unimportant, because the dyeing properties of these man-made fibres are similar to those of wool, and, in admixture with wool, the fibres promote milling shrinkage.

The selection of dyes for inclusion in the mixture was based on the inclusion of an acid dye for protein fibres, a basic dye for Orlon, and disperse dyes for cellulose acetate, nylon, and Terylene. Dyes of suitable colours were chosen after staining tests had been carried out with 1% solutions of some twenty fluorescent dyes in 40% alcohol. In addition, a fluorescent brightening agent was added to provide a sharp distinction between carbonisable and other fibres. Finally, in order to avoid complications due to the presence of fatty matter in the materials tested, a non-ionic detergent was added to the mixture, so that the fibres could be cleaned during the test itself, and the pH was adjusted to give effective discrimination between the three classes of fibre. A lengthy series of trials with various dyes in different proportions and concentrations led to the selection of the following mixture—

TABLE I

Colours observed with Reagent A

Class	Fibre	Colour in Daylight	Colour in Ultra-violet Radiation
Carbonisable	Cotton	Yellowish green	Brilliant blue
	Viscose rayon	Yellowish green	Brilliant blue
	Cuprammonium rayon	Yellowish green	Brilliant blue
	Rayolanda (Courtaulds)	Yellowish green	Brilliant blue
Non-carbonisable	Secondary cellulose acetate	Yellowish green	Green
	Nylon	Pale orange	Deep yellow
	Terylene (ICI)	Very pale yellowish green	Bluish green
	Orlon (DuP)	Yellowish green	Green
Protein	Fibrolane (Courtaulds)	Deep red	Deep red
	Ardil (ICI)	Deep red	Deep red
	Wool	Bluish red	Bluish red

REAGENT A

Duranol Brilliant Yellow 6G 300 powder (C.I. Disperse Yellow 13)	15 ml. of 0·1% soln.
Acronol Yellow TC 180 (C.I. Basic Yellow 1)	10 ml. of 0·1% soln.
Sulphorhodamine B (C.I. Acid Red 52)	15 ml. of 0·1% soln.
Blancol BT (C.I. Fluorescent Brightening Agent 63)	20 ml. of 0·1% soln.
Lissapol N (ICI)	1 ml.
Sulphuric acid (1·0 N.)	1 ml.
Water	138 ml.

When fibres are boiled in Reagent A for 2 min., and then washed thoroughly with water, the colours listed in Table I are obtained, examination under ultraviolet radiation being made with a mercury discharge unit giving 95% of its radiation at 3650 A. If, then, a blend is found to contain fibres which are not coloured red, and which do not give a brilliant blue fluorescence in ultraviolet radiation, it may be concluded that non-carbonisable man-made fibres are present.

The nature of the non-carbonisable fibres is not sharply defined by Reagent A, but they are readily identified by Reagent B, which is prepared by mixing 1% solutions in 40% alcohol of the following compounds in the specified proportions—

REAGENT B

	Parts (vol.)
Duranol Brilliant Yellow 6G 300 powder (C.I. Disperse Yellow 13)	3
Lissamine Flavine FFS (C.I. Acid Yellow 7)	3
Rhodamine B (C.I. Basic Violet 10)	5
Blancol BT (C.I. Fluorescent Brightening Agent 63)	3

When fibres are treated with Reagent B at room temperature for 20 min., and then washed in water, the colours shown in Table II are obtained.

TABLE II

Colours observed with Reagent B

Fibre	Colour in Daylight	Colour in Ultraviolet Radiation
Secondary cellulose acetate	Orange	Deep yellow
Nylon	Greenish yellow	Green
Terylene	White	Blue-violet
Orlon	Pink	Red

DYED FIBRES

In order to discover whether Reagent A could be used successfully with dyed fibres, tests were carried out with dyed samples of viscose rayon, cellulose acetate, nylon, and wool. Lightly dyed materials were readily classified as carbonisable, non-carbonisable, and protein, but the colour of the fluorescence was sometimes different from that obtained with undyed fibres, because the mercury discharge unit gave some visible light. Heavily dyed and dark-coloured materials, however, gave no fluorescence, and it was recognised that stripping would be necessary before dyed materials could be identified. It was still hoped that a superficial strip would be sufficient to permit identification with fluorescent compounds, whereas an almost perfect strip is essential when normal methods of staining are used; and if only mild stripping is required, it should be possible to carry out stripping and staining with a single reagent.

Of the stripping agents available, Formosul (Br) was selected as the most suitable. It was first shown that undyed fibres could be stained satisfactorily with the mixture of Duranol Brilliant Yellow 6G, Acronol Yellow TC, Sulphorhodamine B, and Blancol BT to which Formosul and acetic acid had been added. Among the non-carbonisable fibres, little difficulty was encountered with cellulose acetate and nylon, but Terylene and Orlon were more troublesome. With the latter fibres, improved results were obtained by including a swelling agent, Tumescal D (ICI), in the combined stripping and staining reagent. Unfortunately, an emulsion of Tumescal D must be prepared separately at a temperature above 85°C., and the temperature of the reagent to which it is added must also be 85°C. or above. It was found more convenient, therefore, to use *m*-cresol as the swelling agent, emulsification being brought about with Dispersol A (ICI), which has the advantage of assisting the removal of disperse dyes from cellulose acetate and emulsifying any fatty matter present on the fibres.

The final composition of Reagent C, for use with dyed fibres, was as follows—

REAGENT C

Duranol Brilliant Yellow 6G 300 powder (C.I. Disperse Yellow 13)	15 ml. of 0·1% soln.
Acronol Yellow TC 180 (C.I. Basic Yellow 1)	10 ml. of 0·1% soln.
Sulphorhodamine B (C.I. Acid Red 52)	15 ml. of 0·1% soln.
Blancol BT (C.I. Fluorescent Brightening Agent 63)	20 ml. of 0·1% soln.
Dispersol A	40 ml. of 2·0% soln.
m-Cresol	1 ml.
Acetic acid (30%)	2 ml.
Water	97 ml.
Formosul (C.I. Reducing Agent 2)	2 g.

The reagent is prepared by mixing the solutions of Duranol Brilliant Yellow 6G and Dispersol A and heating until the mixture clears, when the solutions of Acronol Yellow TC, Sulphorhodamine B, and Blancol BT are added. The water and the *m*-cresol are then heated in a separate vessel to give a clear solution, which is added to the warm solution of the dyes. After the mixture has cooled, acetic acid is added, and the volume adjusted to 200 ml. The Formosul is added to the solution just before use, the dyed material under examination being immersed in the cold solution, which is then raised to the boil.

Tests were made on fibres which had been dyed with a wide range of dyes of various types—viz. disperse and basic dyes on cellulose acetate; acid, basic, and disperse dyes on nylon; disperse dyes on Terylene; basic and acid dyes (cuprous ion method) on Orlon; and acid, basic, azoic, and mordant dyes on wool. No special attention was paid to carbonisable fibres, because it is the detection of non-carbonisable fibres which is important. With

pale dyeings on cellulose acetate, nylon, and Orlon the fibres could be identified after 2–15 min. boiling; but 25 min. boiling was needed with Terylene, and with other fibres when they were heavily dyed. Similar results were obtained with the available range of commercial fabrics dyed with unknown types of dye, the non-carbonisable fibres giving a yellowish-green fluorescence.

Cases likely to cause special difficulty were also examined, e.g. heavy dyeings of mordant dyes on nylon and wool, and of azoic dyes on Terylene. When dyed with 8% Solochrome Black WDFA or 6 BN (C.I. Mordant Black 11 or 3), for example, nylon gave no fluorescence after treatment with Reagent C, and only slight fluorescence when dyed with Solochrome Black F or PV 200 (C.I. Mordant Black 5 or 9). In such cases, stripping with a 2% solution of potassium dichromate in 1·0 N. sulphuric acid for 3–4 min., or with a 0·25% solution for 12–15 min., and then boiling for a few minutes in a 1% solution of Irgalon BT (Gy) to remove chromium, gave fibres which, after treatment with Reagent C, showed strong green fluorescence.

In the case of Terylene heavily dyed with azoic dyes, e.g. 6% Dispersol Diazo Black and 6% Brentosyn BB, it was found necessary to boil the material with a solution containing 0·5% sodium chlorite, 3% acetic acid (30%), and 0·2% Tumescal D for 25 min. before sufficient stripping was obtained to allow the fibres to assume a green fluorescence on being treated with Reagent C.

Finally, although the detection of non-carbonisable man-made fibres is the chief concern of this paper, mention should be made of the fact that no fluorescence could be obtained with heavily dyed cotton, especially when Sulphur Blacks had been used, though good results were generally obtained with viscose rayon and Reagent C.

Conclusion

The classification of undyed fibres as carbonisable, non-carbonisable, and protein, and the identification of non-carbonisable fibres, can be carried out easily and quickly with Reagents A and B. Most dyed fibres can be classified and identified with equal certainty by means of Reagent C, which contains Formosul as the stripping agent. When, however, the fibres are heavily dyed, and especially when they are dyed black, more severe methods of stripping must be used before fluorescence can be developed with Reagent C. This is unfortunate, because it means that, when Reagent C fails to reveal the presence of non-carbonisable fibres, supplementary tests must be applied before their absence can be taken as certain. Although staining with fluorescent compounds has not, therefore, proved to be as infallible a test for non-carbonisable fibres as was at first hoped, Reagent C will, it is believed, find considerable use in the low-woollen industry.

* * *

The authors are indebted to the Wool Textile Research Council for financial assistance, and to Dr. C. B. Stevens for a number of helpful discussions.

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(*Received 5th February 1957*)

Reclamation of Fibres from Rags
II—The Isolation of Wool from Mixtures of Wool and Terylene

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Wool may be recovered from mixtures of Terylene and wool by treatment with a 2% solution of hydrazine in butanol at 100°C. for 60 min. Whereas Terylene is completely destroyed under these conditions, the wool suffers only slight damage.

When the different fibres in rags are comparable in value, the ideal method of processing is clearly one in which all types of fibre are separated and recovered for re-use. The non-carbonisable fibres which are commonly mixed with wool have one property in common, viz. a low affinity for water, and any general method of separating wool from blends containing cellulose acetate, nylon, Terylene, and Orlon must be based on this property or on some closely related property, such as ease of electrification by friction[1]. Attempts to devise such a process have so far proved unsuccessful, and until one is available the less desirable method of destroying the non-carbonisable components of a blend in order to recover the wool for re-use must be adopted. The purpose of this investigation was to devise a method of this kind for mixtures of wool and Terylene, a case which has a special interest because of the increasing use of Terylene staple fibre in conjunction with wool, and because of the difficulty of dyeing fabrics made from reclaimed fibres in such a way as to mask the presence of Terylene.

Although a polyester, Terylene shows a surprisingly high resistance to attack by alkalis because of its high crystallinity; attack proceeds from the outside inwards, and a 4% solution of sodium hydroxide causes a loss in weight of only 20% in 80 min. at 100°C. Since wool is completely destroyed under the same conditions, alkalis seem hardly likely to merit serious consideration as a means of isolating wool from mixtures of wool and Terylene. There is, however, one case which deserves special consideration: esters are readily degraded by hydrazine, with liberation of the alcohol and formation of the hydrazide of the acid—

$$R\text{·}COO\text{·}R' + H_2N\text{·}NH_2 \rightarrow R\text{·}CO\text{·}NH\text{·}NH_2 + R'\text{·}OH$$

and Terylene should be attacked rapidly, in spite

of its crystallinity, if the hydrazine molecule is small enough to penetrate the structure and cause general degradation instead of superficial attack. Hydrazine, however, is known to attack the peptide bonds of proteins[3,4], with formation of the hydrazides of the amino acids—

$$-CHR\cdot CO\cdot NH\cdot CHR'- + H_2N\cdot NH_2 \rightarrow -CHR\cdot CO\cdot NH\cdot NH_2 + H_2N\cdot CHR'-$$

and unless reaction with the ester linkage of Terylene proceeds much more quickly than with the peptide bonds of keratin, there can be no hope of recovering wool from mixtures of Terylene and wool by destroying the former with hydrazine. The purpose of this investigation was, therefore, to study the action of hydrazine on wool and Terylene in order to define the optimum conditions for preferential attack on Terylene.

Experimental

MATERIALS

(*a*) HYDRAZINE—Anhydrous hydrazine (99·2%), supplied by Whiffen & Sons Ltd., was used without further purification.

(*b*) WOOL—A 1/16s worsted yarn, made from

TABLE I

Effect of Butanolic Hydrazine

Concn. of Hydrazine (% by vol.)	Time of Treatment (min.)	Wool Extension at Break (%)	Wool Breaking Load (% of original)	Wool S.E.* (%) of Breaking Load	Terylene Extension at Break (%)	Terylene Breaking Load (% of original)	Terylene S.E.* (%) of Breaking Load
		TREATED SEPARATELY					
—	—	33·9	100·0	2·0	34·0	100·0	1·8
1	5	40·1	107·4	2·4	33·1	90·0	2·5
2		42·7	117·3	2·5	18·3	40·7	4·5
3		—	—	—	12·5	30·5	2·3
4		—	—	—	4·2	8·4	17·7
5		37·3	104·8	1·9	2·1	2·3	18·5
10		34·7	97·8	2·9	—	—	—
1	15	39·5	99·3	3·4	28·9	71·1	2·8
2		39·6	109·5	2·1	11·0	28·2	4·6
3		—	—	—	5·0	9·5	12·9
4		—	—	—	2·4	2·0	13·3
5		39·7	109·0	2·0	—	—	—
10		37·5	105·1	3·2	—	—	—
1	30	40·0	114·5	2·3	19·1	42·5	2·7
2		38·7	114·4	3·3	5·4	8·8	6·1
3		37·2	107·9	1·7	—	—	—
4		40·3	109·5	1·8	—	—	—
1	45	39·7	105·1	3·4	11·6	26·0	2·8
2		40·0	102·9	2·1	2·7	3·8	3·9
3		43·1	101·5	1·6	—	—	—
4		37·7	85·8	3·8	—	—	—
1	60	42·9	105·8	2·8	9·9	23·3	2·6
2		39·4	99·3	3·0	1·3	0·9	8·8
3		39·1	95·0	2·0	—	—	—
		TREATED TOGETHER					
1	5	41·2	106·8	4·4	32·3	86·1	4·6
2		38·5	108·6	2·6	28·4	73·1	3·9
5		36·3	98·9	5·6	5·8	12·6	3·0
10		40·5	83·2	2·4	—	—	—
1	15	42·7	109·3	2·2	31·3	78·1	2·9
2		37·8	110·6	3·1	17·4	38·3	3·0
3		41·6	109·1	2·7	6·9	13·3	2·6
4		45·4	103·6	1·1	3·8	3·3	7·2
1	30	41·8	103·3	4·3	25·4	55·5	3·6
2		40·1	107·8	2·6	7·2	15·6	2·6
3		42·1	100·8	3·4	1·8	1·3	5·0
4		40·9	90·5	2·3	—	—	—
1	45	40·5	106·6	3·1	13·9	33·5	2·1
2		42·6	108·2	2·3	3·7	3·3	6·1
3		40·0	100·2	3·1	—	—	—
4		36·2	94·5	2·9	—	—	—
1	60	41·0	110·9	3·0	10·9	28·0	2·6
2		40·5	105·0	3·1	1·5	0·9	17·2
3		36·1	94·7	3·7	—	—	—

* Standard error

64s merino wool, was purified by extraction with alcohol and ether in a Soxhlet apparatus. The yarn was then washed in distilled water, before being conditioned in the humidity room (65% R.H., 22·2°c.).

(c) TERYLENE—A spun yarn (1/13·5s worsted count), composed of 4-denier filaments, was purified in the same way as the worsted yarn.

Procedure and Results

1. TREATMENT OF YARNS WITH SOLUTIONS OF HYDRAZINE IN BUTANOL

Semi-quantitative experiments were carried out in which the worsted and Terylene yarns were treated separately and together with solutions of hydrazine in order to compare the rates at which the two types of fibre are attacked. No special sampling procedure was adopted, successive lengths of the purified and conditioned yarns being taken and immersed in an anhydrous solution of hydrazine in *n*-butanol (25·0 ml.) at 100°c. Air bubbles were removed by stirring, and after the desired time of treatment the yarn was removed, washed twice with cold ethanol, and thoroughly washed with water before being conditioned in the humidity room. When the worsted and Terylene yarns were treated separately, 0·25-g. samples were used; and when they were treated together, 0·125-g. samples of each.

The extension at break and the breaking load of the conditioned yarns were determined with a Scott inclined-plane tester, ten tests being made on the separately treated yarns and seven tests on each of the yarns treated together. The test-length was 6 in. in all cases, and the results are given in Table I, illustrated by Fig. 1.

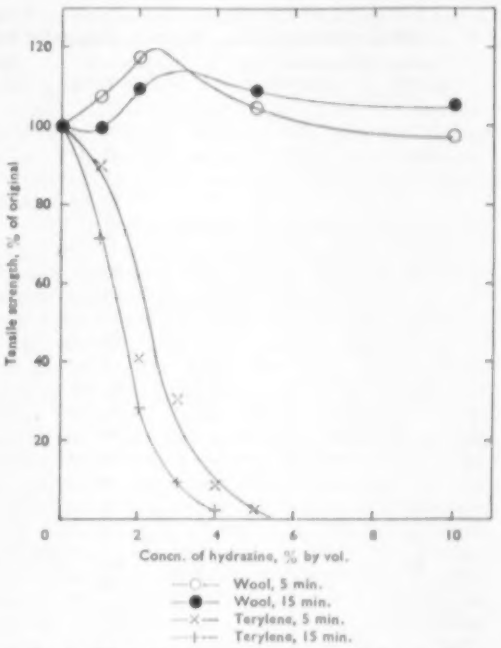

FIG. 1—Effect of Butanolic Hydrazine on Tensile Strength of Worsted and Terylene Yarns treated separately

2. TREATMENT OF YARNS WITH SOLUTIONS OF HYDRAZINE HYDRATE IN BUTANOL

As anhydrous hydrazine is expensive, the possibility of using hydrazine hydrate ($H_2N{\cdot}NH_2{\cdot}H_2O$) was next examined. The procedure was exactly the same as in the preceding experiments, but only those concentrations of hydrazine hydrate were used that might be expected, in the light of the above results, to cause severe degradation of Terylene. The results are given in Table II.

3. TREATMENT OF YARNS WITH SOLUTIONS OF HYDRAZINE IN MIXTURES OF BUTANOL AND WATER

Because of the success of the experiments with hydrazine hydrate, it was decided to examine the possibility of decreasing the cost of the reagent still further by using mixtures of butanol and water as the solvent.

In these experiments the worsted and Terylene yarns were sampled according to the following strict procedure. A number of lengths of yarn, equal to the number of experiments to be performed, were drawn off the bobbin, the procedure being repeated ten times in succession. Thus ten samples, one from each draw, were used in each experiment. After purification and conditioning in the usual manner, equal weights of wool and Terylene were treated together with the appropriate solutions for the desired time at 100°c. The solutions were prepared by adding hydrazine or aqueous solutions of hydrazine to butanol to give the required concentration of hydrazine in the reagent, and the properties of the worsted yarn, after treatment, are given in Table III; the Terylene was completely degraded in all cases.

The results are illustrated by Fig. 2, from which it is evident that the lowest concentrations of hydrazine in the aqueous solutions used in making

TABLE II

Effect of Butanolic Hydrazine Hydrate

Concn. of Hydrazine Hydrate (% by vol.)	Time of Treatment (min.)	Wool Extension at Break (%)	Wool Breaking Load (% of original)	Wool S.E. (%) of Breaking Load	Terylene Extension at Break (%)	Terylene Breaking Load (% of original)	Terylene S.E. (%) of Breaking Load
			TREATED TOGETHER				
7·81*	5	42·8	99·1	1·9	5·8	9·9	6·0
6·25*	15	39·5	101·7	3·5	2·2	1·1	7·2
4·69*	30	43·8	101·7	1·6	2·3	1·3	8·9
3·13*	45	39·0	96·8	2·0	3·4	2·8	9·2
3·13*	60	41·8	98·1	1·6	1·7	1·0	6·3

* Corresponding to 5, 4, 3, and 2% of hydrazine

TABLE III

Effect of Hydrazine in Aqueous Butanolic Solution on Wool treated in presence of Terylene

Concn. of Hydrazine Aq. Soln. (% by vol.)	Reagent	Time of Treatment (min.)	Extension at Break (%)	Breaking Load (g.)	Breaking Load (% of original)	S.E. (%)
—	—	—	33·3	404	100·0	2·6
100	2	60	42·3	569	140·8	2·0
	3	45	43·0	546	135·1	2·7
	4	30	39·6	549	136·0	3·3
	5	15	42·5	572	141·7	3·5
	10	5	34·6	475	117·5	2·8
80	2	60	43·9	566	140·0	3·0
	3	45	41·9	533	132·0	3·1
	4	30	40·6	536	132·6	1·8
	5	15	41·9	517	128·0	1·5
	10	5	29·3	359	88·9	3·3
64	2	60	43·6	544	134·5	3·5
	3	45	44·0	528	130·6	3·1
	4	30	33·8	437	108·3	2·3
	5	15	36·4	464	114·7	3·1
	10	5	31·7	325	80·5	1·3
58	10	5	30·4	286	70·8	3·0
50	10	5	19·4	175	43·3	9·7
40	2	60	39·8	527	130·4	2·8
	3	45	33·3	423	104·6	2·5
	4	30	22·4	331	82·0	2·2
	5	15	25·2	323	79·9	3·7
	10	5	Too weak to measure			
30	4	30	23·3	195	48·2	2·6
	5	15	Too weak to measure			
25·6	2	60	27·8	409	101·3	2·8
	3	45	10·5	78	19·4	17·8
	4	30				
	5	15	Too weak to measure			
	10	5				

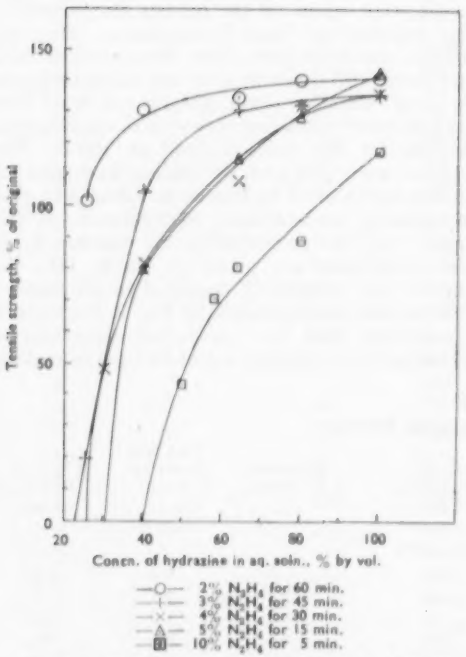

FIG. 2—Effect of Hydrazine in Aqueous Butanolic Solution on Worsted and Terylene Yarns treated together

up the reagents, and the associated times of treatment, must be as specified in Table IV if the strength of the worsted yarn is to remain unaltered.

TABLE IV

Concn. of Hydrazine Aq. Soln. (% by vol.)	Reagent	Time of Treatment (min.)
85	10	5
54	5	15
52	4	30
40	3	45
25	2	60

4. TREATMENT OF WOOL FIBRES WITH SOLUTIONS OF HYDRAZINE

It is well known that the strength of a yarn is not a reliable guide to the extent to which the component fibres may have been damaged in any treatment, because any alteration in friction, due to superficial attack, changes the proportions of the fibres which break and slip when the yarn is ruptured. Further tests were made, therefore, to determine the extent to which the properties of single wool fibres were altered by treatment with hydrazine.

Staples of Leicester wool were purified by extraction with alcohol and ether in a Soxhlet apparatus, followed by washing in distilled water. The root ends of non-medullated fibres were used, several 5-cm. lengths being attached to light

glass hooks so that their load–extension curves could be determined up to 30% in distilled water at 22·2°C. After calibration in this way, the fibres were allowed to rest in water for 24 hr. before being dried in room air. In each experiment six of the calibrated fibres were treated with a solution of hydrazine (25 ml.) for the desired time at 100°C. in the presence of wool (0·125 g.) and Terylene (0·125 g.). After being washed in running water, the fibres were restretched so that the change in resistance to 30% extension (C.R.E.), due to the treatment, could be calculated. The results are given in Table V, which includes data for the C.R.E. due to treatment with butanol alone, as well as the C.R.E. due to a second extension of an untreated fibre in water.

TABLE V

Concn. of Hydrazine Aq. Soln. (%, by vol.) Reagent	Time of Treatment (min.)	Extension at Break (%)	C.R.E. (%)
100* 2	45	48·8	8·4
2	60	48·9	12·1
10	5	53·7	24·8
64 2	45	56·0	15·0
— 0†	5	54·4	5·8
— 0†	60	54·2	9·3
— —	—	—	1·5‡

* Anhydrous
† Butanol alone
‡ Second extension of untreated fibre in water

Discussion

It is evident from the results of Table I and Fig. 1 that Terylene is much more rapidly degraded than wool by solutions of hydrazine in butanol at 100°C., complete destruction being given by 2, 3, 4, 5, and 10% solutions in 60, 45, 30, 15, and 5 min. respectively. The worsted yarn, treated under the same conditions, is stronger than the untreated yarn in most cases, and only very slightly weaker when the 2% and 10% solutions are used. Similarly, when equal weights of Terylene and wool are treated together, the Terylene is destroyed completely by all the solutions before the worsted yarn suffers any serious loss of strength.

In corresponding concentrations, hydrazine hydrate seems to be just as effective as hydrazine in attacking Terylene, and the worsted yarn is again almost unaltered in strength (Table II). It would, of course, be an advantage, as regards cost, if aqueous solutions of hydrazine could be used in preparing reagents for treating mixtures of Terylene and wool, and it is shown in Table IV that a 2% solution of hydrazine, prepared by adding a 25% (by vol.) solution in water to butanol, causes complete destruction of Terylene in 60 min. without reducing the strength of the worsted yarn. The water content of the mixture is, however, only 6%, and the cost of the reagent remains high.

It will be noted that the strength of the worsted yarn, after treatment, is in many cases (Tables I, II, and especially III) higher than that of the original yarn. This is an indication that there has been some attack on the fibres, the surface friction being increased, and a greater proportion of the fibres broken when the yarn is ruptured. Support for this deduction is given by the data of Table V, single fibres being weakened under conditions which give a stronger yarn. From the results obtained with the 2% solution of hydrazine, one prepared from anhydrous hydrazine and the other from a 64% solution in water, it is obvious that the presence of water in the reagent is deleterious to the wool. In addition, the results show that wool suffers less damage when treated with a 2% solution of anhydrous hydrazine in butanol for 60 min. than with a 10% solution for 5 min. The C.R.E. due to the treatment is only 12·1 − 1·5 = 10·6%, and of this a high proportion is not due to hydrazine, as may be seen from the data for fibres treated with butanol alone. It may be concluded, therefore, that the best conditions for recovering wool from mixtures of wool and Terylene are treatment with a 2% solution of hydrazine in butanol at 100°C. for 60 min.

* * *

The authors are indebted to the Wool Textile Research Council for grants in aid of this investigation, and to Messrs. Whiffen & Sons Ltd. for gifts of chemicals.

TEXTILE CHEMISTRY LABORATORY
DEPARTMENT OF TEXTILE INDUSTRIES
THE UNIVERSITY
LEEDS 2

(*Received 19th February 1957*)

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[3] Akabori, S., Ohno, K., and Narita, K., *Bull. Chem. Soc. Japan*, **25**, 214 (1952).
[4] Blackburn, S., and Lee, G. R., *J. Textile Inst.*, **45**, T 487 (1954).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Johann Peter Griess

Having had the good fortune to be able to throw some light on the affairs of the firm of Perkin & Sons [1], I am now anxious to learn what happened to the family of J. P. Griess, of diazo fame, who died in 1888 and left four children.

I should be very grateful if any reader who may have special information or documents relating to this other pioneer of synthetic dyes would communicate with me.

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15th June 1957

[1] Cliffe, W. H., J.S.D.C., **73**, 312 (July 1957).

Effect of Concentrated Sulphuric Acid on the Various Constituent Amino Acids of Wool

As a preliminary to an investigation of the effect of the wool carbonising process upon the various membranes of the wool fibre [1], a study has been made of the effect of concentrated sulphuric acid on the amino acids known to occur in wool.

The individual amino acids were treated with concentrated sulphuric acid at 29°C. for 7 days. Any solid products were isolated by pouring the solutions into ether and filtering off the solids formed. Residual acidity of the solid products was removed by dissolving them in water and neutralising with solid barium carbonate, the product finally being isolated by evaporation of the filtrate.

The various products of the reactions were each compared chromatographically and electrophoretically with the corresponding untreated amino acids as follows—

(*a*) Ascending-type chromatograms were prepared on Whatman No. 1 paper using phenol–water and butanol–acetic acid–water solvents.

(*b*) Electrophoresis was carried out in an EEL X 118 electrophoretic apparatus. A borate buffer (pH 8) was used, and a current of 1·5 ma. was passed for 16 hr.

Amino-acid spots were developed with an acetone solution of ninhydrin.

The constituent amino acids of wool were found to be unchanged except for the following—(1) phenylalanine, (2) tyrosine, (3) serine, (4) threonine, (5) tryptophan, (6) arginine.

(1) Phenylalanine reacted completely under the above conditions. Chromatographic examination indicated one large (*b*) and one subsidiary spot (*a*) (Table I). Electrophoresis indicated that the products were as strongly acidic as aspartic acid.

The major product recrystallised from water as flat triangular plates of a monohydrate. After dehydration *in vacuo* the following analysis figures were obtained: C 41·2%, H 4·56%, N 5·72%, S 13·1%. For comparison, the calculated monosulphonated values for phenylalanine are: C 41·4%, H 4·28%, N 5·99%, S 13·7%.

Infrared examination of this compound showed absorption peaks consistent with the presence of a sulpho group and a *para*-substituted benzene ring. The acid was stable in aqueous solution, but prolonged (24 hr.) boiling with 6 N. hydrochloric acid converted it back to phenylalanine.

(2) The product isolated after treating tyrosine with concentrated sulphuric acid, when examined chromatographically, was found to give one main spot and one subsidiary spot. The substances causing these spots were stable to mild hydrolysis, and therefore were not simply sulphate esters formed with the hydroxyl group of tyrosine. By analogy with phenylalanine the products are probably sulphonated derivatives. Prolonged (24 hr.) boiling with 6 N. hydrochloric acid reconverted the derivatives to tyrosine. This reaction is closely analogous to reactions of benzenesulphonic acids.

Infrared examination again indicated the presence of sulphonic acid groups in the products, but as the latter were not separated, the number and position of such sulpho groups in the phenyl groups of the tyrosine molecule could not be determined.

(3) Serine was converted by concentrated sulphuric acid into a derivative which, when examined electrophoretically, proved to be acidic in nature. As both the amino and carboxyl groups appear to be unchanged, the product is presumably the acid sulphuric ester of the free hydroxyl group, the structure being $HO{\cdot}SO_2{\cdot}O{\cdot}CH_2{\cdot}CH(NH_2){\cdot}COOH$.

The presence of a sulpho group was again confirmed by infrared examination. The product is rapidly hydrolysed by refluxing with 6 N. hydrochloric acid for 30 min., or more slowly on standing in water at room temperature for 7 days. By this method of hydrolysis the sulphate is reconverted to serine.

(4) Threonine gave, with concentrated sulphuric acid, mainly the unchanged amino acid; but another minor spot occurred on the chromatogram. When examined electrophoretically this minor product was found to be acidic in nature, and was readily hydrolysed to threonine on standing in water for 12 hr. or on being refluxed with 6 N. hydrochloric acid for 1 hr. The unknown spot on the chromatogram is therefore probably due to the acid sulphuric ester of threonine.

(5) Tryptophan gave two derivatives, which when examined electrophoretically were both found to be acidic in nature. The chromatographic R_f values were similar to those of sulphonated phenylalanine and tyrosine, and these are probably again compounds sulphonated in the indole nucleus of the tryptophan.

(6) Arginine, when treated similarly to the other amino acids, was mainly unchanged, but two new spots appeared on the chromatogram. These

were identical with those formed by citrulline and ornithine.

The R_f values in phenol–water for the new sulphonated products are given in Table I.

TABLE I

Phenylalanine	...	(*a*)	0·13	(*b*)	0·23
Tyrosine ...	...	(*a*)	0	(*b*)	0·095
Serine ...	...		0·15		
Threonine ...	...		0·064		
Tryptophan	...	(*a*)	0	(*b*)	0

The above evidence supports the work of Lustig and Kondritzer [2], who found that red hair, after treatment with concentrated sulphuric acid, showed losses of the following amino acids—phenylalanine, tyrosine, serine, tryptophan, and arginine. They explained these results by suggesting that sulphonation of the benzene ring could have occurred in the appropriate compounds.

Work is now being carried out to isolate the above hitherto unknown amino-acid derivatives from wool which has been treated with concentrated sulphuric acid.

Grateful acknowledgment is made to Mr. J. Keighley for the infrared measurements. We also wish to thank the Wool Textile Research Council for permission to publish this work and for a grant to one of us (D. H. R.).

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8th July 1957

[1] Elliott and Roberts, J.S.D.C., **72**, 370 (1956).
[2] Lustig and Kondritzer, *Arch. Biochem.*, **8**, 51 (1938).

Notes

Meetings of Council and Committees
August

Council— No meeting

Colour Index Editorial Panel— 8th and 22nd

Fastness Tests Co-ordinating— 28th

Abstracts Subcommittee— 13th

Perkin Centenary Awards

The Perkin Centenary Trust has awarded the Perkin Centenary Fellowship to Mr. John Edward Bloor of Manchester for study in the Department of Chemistry, Manchester College of Science and Technology, on the structure and properties of *mero*cyanine dyes and related compounds.

Perkin Centenary Scholarships have been awarded to Mr. James McCartney of Newtownards, Northern Ireland, tenable at Queen's University, Belfast; to Mr. Clive Milne of East Ardsley, near Wakefield, Yorkshire, tenable at Bradford Institute of Technology; and to Miss Gabrielle Griffin of Withington, Manchester, tenable at Manchester University.

The Perkin Centenary Trust has received or been promised some £36,000 so far. The Trustees have announced a number of awards to promote technical education in any aspect of the manufacture or the application of colouring matters.

The Trustees are Mr. H. Jackson (Chairman), Dr. D. W. Hill, Mr. W. R. Mathers, Mr. M. W. Perrin, and Sir Robert Robinson. The Secretary to the Trustees is Mr. J. R. Ruck Keene, to whom enquiries relating to the awards should be addressed, c/o The Chemical Society, Burlington House, London W.1.

London Section Perkin Centenary Prize Essay Competition

The response to this competition was disappointing, and the London Section Committee has decided that no entry was received of sufficient merit to warrant the award of the full prize. A lesser award of £5 5s. 0d. has been awarded to Mr. R. L. Jones of Cwmbran, Monmouthshire, for his essay *The Influence of Perkin's Discovery on the Textile Dyeing and Finishing Industry.*

Fifth London Lecture

This will be given by Miss Anne M. Buck, Keeper of the Gallery of English Costume, Platt Hall, Rusholme, Manchester, and will be entitled *The Art of Costume*. It will be held on 11th October 1957 at the Waldorf Hotel, London W.C.2, at 7 p.m.

New Books and Publications

Handbuch für die textile Nassveredlung
Ein Nachschlagewerk mit Tabellen und Formeln für die Praxis

By Gerhard Melzer. Pp. 394. Berlin: VEB Verlag Technik 1956. Price, DM 23.00.

Yet a further contribution to the already voluminous German textile literature! It is intended to help the technologist in his struggle to keep abreast with the ever growing advances in the field of dyeing and finishing. Herr Melzer's subtitle means "a book of reference with tables and recipes for practical use", and this description is most appropriate. In truly remarkable fashion the author gives a bird's eye view of the multitudinous operations generically referred to as the "wet processing of textiles". He makes a point of

giving full working instructions for all the different processes described, thereby providing answers to the many problems met with in day-to-day practice.

The book is divided into five chapters. The first, comprising 90 pages, is termed *Textile Technology*, and presents a survey of natural and man-made fibres, and their physical and chemical properties. The second chapter, with 76 pages, deals with the processes carried out preparatory to dyeing, and also with conventional finishing methods. Chapter 3, with 67 pages, is concerned with dyeing; whilst Chapter 4, the longest in the book, with 115 pages, deals with the laboratory control of processing. A fifth and final chapter, of 12 pages, contains miscellaneous information, and this is followed by a list of dyemakers and producers of textile auxiliaries. A short bibliography (all German references) is followed by a very comprehensive subject-index (26 pages).

As already said, the author has set out to provide the dyeing and finishing technologist with a modern reference book, kept to manageable proportions. Taken by and large, this has been achieved, and the good features of the book far outweigh its shortcomings. What, then, are the advantages? Perhaps the biggest is the fact that its pages are packed with readily accessible information, which whenever possible is presented in tabular form, set out in neat and clear type. The style is frequently telegraphic, but loses nothing by this. The very adequate subject-index has already been referred to, and a further feature is the provision of "key words" down the margin of each page, which greatly facilitates the quick location of information. The working instructions are clear and concise, forming one of the best features of the book, along with the sections dealing with fibre and dye identification, and analysis of textile auxiliaries.

Now the shortcomings. First and foremost, this book has been written for the German (and possibly Swiss) technologist. This in itself may not be a shortcoming, but it develops into one if the book is offered for sale further afield. Thus practically all the commercial products referred to, be they dyes, sizes, bleaches, resins, finishing agents, or auxiliaries, are of German or Swiss manufacture. The reviewer is not informed whether or not the products of French, British, American, etc. dye and auxiliary manufacturers are freely available in Eastern Germany. If not, it is obviously understandable that Herr Melzer has decided not to include them. However, he must not be surprised if his book does not find the appeal here that it would otherwise deserve.

Another point of criticism is the reproduction of tables from other sources, without sufficient discrimination or checking. Thus on p. 17 cotton from Ecuador, Pernambuco, and Guayaquil is stated to be of North American origin, whilst Northern Ireland does not even get a mention amongst world flax growers (p. 22). It appears that no knowledge exists in Eastern Germany as to whether Acrilan is produced in staple or filament form (p. 40), and on p. 42 Imperial Chemical Industries Ltd. are quoted as producers of nylon fibres. The only other time ICI is referred to is on p. 210, as producers of water-soluble dyes for cellulose acetate (Solacets). Even so, this firm occupies a privileged position, as no other British, French, or American dye manufacturer is considered worthy of inclusion.

The actual misprints are few. Alkylaryl (p. 101) is mis-spelt; there should be no acidic hydrogen atom in sodium palmitate (formula on p. 271); and the formula of arachidic acid (p. 273) requires amendment too. The note on the stripping of Naphtol AS (p. 189) has inadvertently got mixed up with the Phthalogens.

Certain aspects of dyeing and finishing have not been adequately covered. The dyeing of polyester fibres and fabrics has been completely omitted, and the sections dealing with the production of water-repellent and flame-retardant finishes belie the book's claim to be up to date.

The fact that more space has been devoted to shortcomings than to praise (the author has himself asked for critical observations and suggestions) does not detract from the value of the book. It is a recommendable purchase for all concerned with the practical aspects of dyeing and finishing.

G. W. Madaras

Mechanisch-technologische Textilprüfungen

Teildruck T 14 aus Handbuch für Textilingenieure und Textilpraktiker

By Erich Wagner. 7th edition 1957. Pp. viii + 228. Wuppertal-Elberfeld: Dr. Spohr-Verlag. Price, DM 15.00.

As part of a proposed collection of about fifty books by various authors intended to form a general survey of technical textile subjects, this book deals specifically with the testing of the mechanical properties of textiles from the raw material to the finished product. Although small in size, being intended as a handy reference on the desk of the practical technologist or for slipping into the pocket of the student, its contents are equal in extent to those of most books of a larger format. This has been achieved by the use of comparatively small but clear print, and economy of space has been obtained by suitable arrangement, the use of small line sketches to illustrate the fundamental features only of the types of apparatus used in this branch of technology, and by confining literature references to bare essentials.

Apart from the possible exception of those forms of testing which may apply only to specialised branches, the whole field of the subject is up to date and well covered, the most notable exception being the failure to mention the property of "sewability", a characteristic which has assumed prominence owing to the development of various types of stabilised finishes. Testing for slipping or fraying is but scantily treated and confined to the "sewn-seam" method.

The importance of standard atmospheric conditions and the necessary basic statistical assessment of results are stressed by bestowing upon them prominence of position at the beginning, and not

appending them as an afterthought, as is so often the case.

The usual internationally accepted and tentative methods, as well as national standard methods, are well described, but the reader is required to refer to the corresponding specifications for detailed and comprehensive treatment. In addition to these well established methods, many of the familiar forms of quality control and various attempts to assess "wearability" or "serviceability" are described, but we find little or nothing that is new. Considering the vast range of variables in this field, from the corduroy breeches of a hirsute navvy to the delicate lingerie of a satin-skinned débutante, it is not surprising that no single method exists at the stage of universal acceptance. As the author points out, specialised treatments for particular requirements are essential in this branch of testing if any worthwhile results are to emerge.

Though this book will appeal particularly to the student, the practical technologist will find a use for the various formulae, graphs, and tables, and the book should serve as a fairly comprehensive reference in a convenient form. The monograms would require enlarging to be of any practical value.

There is little that calls for serious adverse criticism, and what little there is will naturally depend upon the views of the individual reader. One might have expected, after reading on p. 47 that the Tex system of yarn counting had superseded the Grex or Decimel system, that the example on the following page as well as subsequent examples would not have been given in the decimal form.

The method of withdrawal of yarn from weft cops for purposes of yarn twist testing, as described on p. 65, should not necessarily be standardised in this form. The manner of withdrawal obviously depends upon the information being sought. The statement which appears on p. 70, to the effect that the tensile strength of a woven fabric in the warp direction *never* yields a result which is 100% of the sum of the strengths of the component warp threads, is not borne out by experience, and, in fact, the very first item in the table on the facing page gives ratios of 0·50–1·02.

Allowing for slight criticism of this nature, it is obvious that the author is fully conversant with the subject-matter, which reveals a wide experience and is clearly the result of intensive research.

The publishers are to be commended on the manner of presentation. One feature of the publication which exemplifies the typical thoroughness usually associated with German productions is revealed by the type of binding which has been used. Most British publishers would have missed the point completely and provided a highly glazed backing. Not so in this case, for the cover of this book has a somewhat rough surface, no doubt intended to serve as a practical example of the effects of abradant materials on the already threadbare pockets of the impecunious student, an example which could possibly propagate the germ of the idea which might eventually lead to a solution of the problems associated with wearability tests.

J. Wood

Titration in Non-aqueous Solvents

By A. H. Beckett and E. H. Tinley. 2nd edition 1957. Pp. iv + 43. Poole: British Drug Houses Ltd. No price.

Those who are familiar with the first edition of this booklet will find no major alterations in the present edition. A few more pages have been added, which include extensions of some sections, a few new sections and diagrams, and a substantial increase in the number of references. It is gratifying to note that the section dealing with solvents has been extended. The more important new additions are sections dealing with glycol–alcohol solvents and high-frequency titrations, although the latter is totally lacking in detail.

For those who are not familiar with the first edition or who are unacquainted with the subject of non-aqueous titrations the book contains much useful information written in simple language. It contains 97 references, about a fifth of which are within the last two years. Too much cannot be expected in a publication of this kind which can be obtained gratis; in spite of this, however, the authors have attempted to cover the more important aspects of the subject. Most of the information is given in general terms or by reference, but in some instances adequate experimental detail is given. Examples of the latter are some solvent purifications, preparation and standardisation of titrants, and determination of amino acids and halide salts.

The theoretical aspect is dealt with somewhat summarily, but the main chapters dealing with solvents, titrants, and the determination of specific classes of compounds are comprehensive in range if not in detail.

The reader of scientific publications undoubtedly wishes to know how up to date is the subject-matter, and should not have to make deductions from references or decode dates which have been put in an unusual place. It is preferable to put the date in conventional form on the title-page. Apart from this minor criticism, the book is a useful publication which should find a place on the bookshelf of anyone interested in analytical chemistry.

W. Armfield

Das Farbensehen

By S. W. Krawkow. Pp. xi + 168. Berlin: Akademie-Verlag 1955. Price, DM 16,00.

This monograph is a translation by P. Klemm of a posthumous work written in 1950 by S. W. Krawkow of the Department of Psychology in the Philosophical Institute of the Academy of Sciences of the U.S.S.R.

It gives a detailed and mainly non-mathematical description of the subject of colour vision, including discussions of the physiological and the subjective aspects of colour perception and colour mixing, and of the phenomena of colour blindness and various manifestations of abnormal colour vision,

including the influence of artificial stimulation, e.g. certain drugs, upon the response of the eye. It will be found especially useful by specialists in the subject for its many references to recent Russian work which would otherwise be difficult of access— the references in fact include 118 to Russian literature sources, but only 69 to those of other countries.

In his introduction the author links the subject to the dialectical materialist ideology of Lenin.

Typography, illustration, and binding are up to the high standards we expect of German scientific publications.

C. H. GILES

Farbmetrik

(Scientia photographica Band 1)

By Hans Arens. 2nd edition 1956. Pp. xi + 83. Berlin: Akademie-Verlag. Price, DM 13.80.

Not many books on the measurement of colour were available in Germany in 1945. This monograph was written chiefly for the benefit of those engaged in colour photography in order that they might obtain a grasp of the fundamental ideas on colour measurement. Physiology, colour vision, etc. are excluded. This second edition differs from the first only in the revision of colour terms and symbols, which are similar to those agreed by the Commission Internationale de l'Éclairage (C.I.E.).

The first chapter discusses some general properties of colour. The second chapter, which occupies the greater part of the book, deals with colorimetry; and difficulties associated with relating objective colour measurements to visual sensations are discussed. The remainder of the book includes a summing up and a list of definitions of colour terms.

Colour is considered both subjectively and objectively, and the C.I.E. system of colour measurement based on the additive mixing of three primary colours is explained. Other systems are also mentioned. Essential facts only are included, and these need to be supplemented by further reading if more detail is required. Comparatively few references are given throughout the book, and such well known names as Hardy and Judd are very briefly mentioned in a reference dated 1940. Most of the references which do occur are to German publications.

The nature of the information given requires a better selection of up-to-date references if it is to be of real use to the reader. These might well have been included in the second edition together with appropriate alterations to the script.

The book is well produced, and the diagrams are very clear. By present-day standards the price is reasonable.

J. C. GUTHRIE

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I— PLANT; MACHINERY; BUILDINGS

PATENTS

Grinding Equipment for Organic Compounds
FBy *BP* 777,605

In conventional machines, edge-runner mills, hammer mills, pin mills, etc., used for grinding organic compounds, hard deposits may sometimes form. If the parts of the mill which perform the comminution are made of slightly elastic materials such as polyamides or polyester–*iso*cyanate copolymers, slight relative movement occurs between these parts and any deposit which forms, so that the latter soon peels off. E.S.

Pelleting Carbon Black
Phillips Petroleum Co. *BP* 775,378

Apparatus in which water is sprayed into the hot carbon black in the mill as it is being pelleted to yield pellets containing < 1% moisture. C.O.C.

Eliminating Entrapment of Air in Yarns Impregnated with Liquid Resin
Specialities Development Corpn. *BP* 773,626

The yarn passes through a chamber whose entry and exit are sealed by the liquid resin. Suction is applied to the chamber to evacuate air entrapped in the yarn by the resin. C.O.C.

Striping Fabric
Springs Cotton Mills *USP* 2,737,042

Apparatus for carrying out the process described in *USP* 2,733,974 (J.S.D.C., **73**, 409 (Aug. 1957)). C.O.C.

Wet Treatment of Cloth
E. Kusters *BP* 773,744

In a machine in which the cloth passes round a roller part or all of which is in the liquor, the surface of the roller is either covered with a resilient porous material capable of taking up the liquor or allowing it to pass through it into the interior of the roller. The roller is mounted eccentrically so that it vibrates as it rotates thus ensuring flow of the liquor relative to the fabric backwards and forwards from one surface to the other of the fabric as it travels with the roller. Thus ensures thorough and even treatment. C.O.C.

Fluid Bed Drier for Cloth
British Rayon Research Assoc. *BP* 773,598

The wet cloth is passed vertically through a bed of hot solid discrete particles, e.g. sand, while hot gas is passed up through the bed. The size and weight of the particles and the velocity and nature of the gas are so chosen that the force exerted by the gas counterbalances the effect of gravity on the particles so as to allow movement of the particles without causing them to be carried away by the gas. C.O.C.

Continuous Drying of Cloth, etc.
J. Dungler *BP* 773,606

The cloth is subjected to the simultaneous action of a current of hot gas and radiation emitted by radiant elements. The radiant elements are raised to their operating temperature solely by contact with the hot gas. This enables the radiant elements to be very small and so able to be placed in the optimum position for effective action on the cloth. C.O.C.

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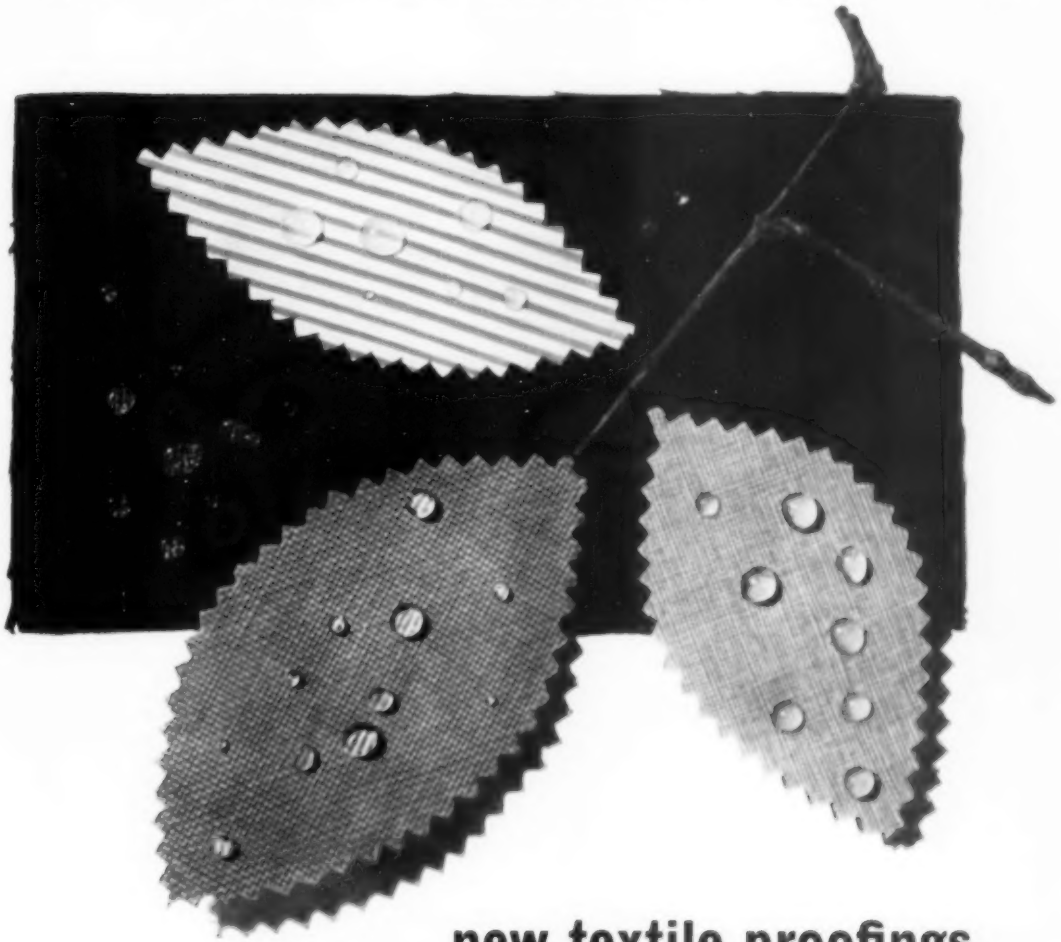

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Inns and the Textile Trade

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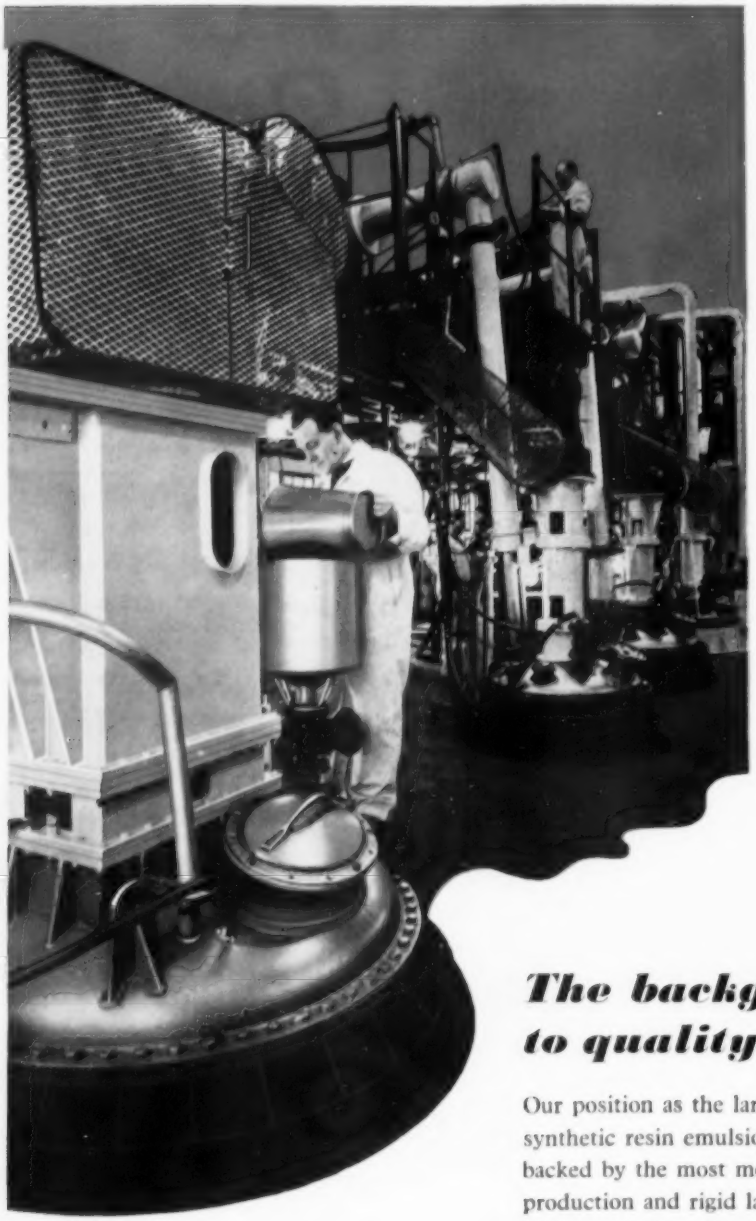

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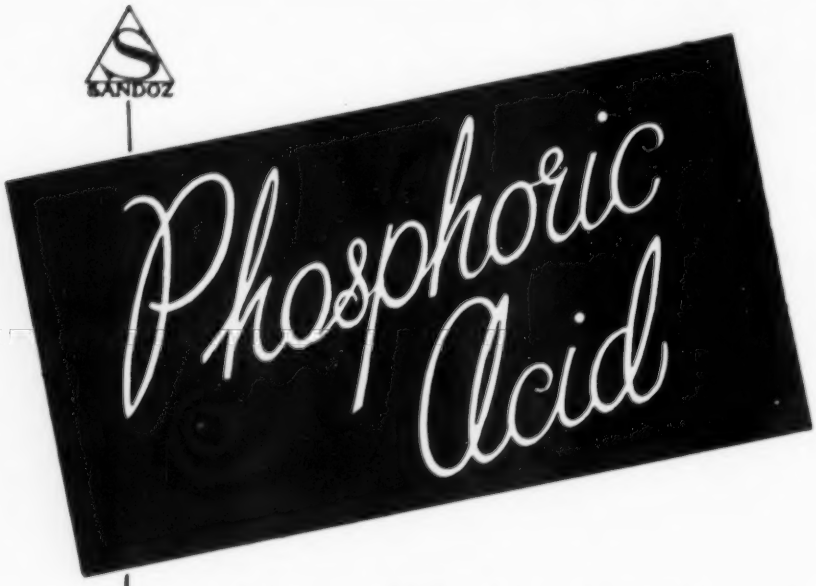

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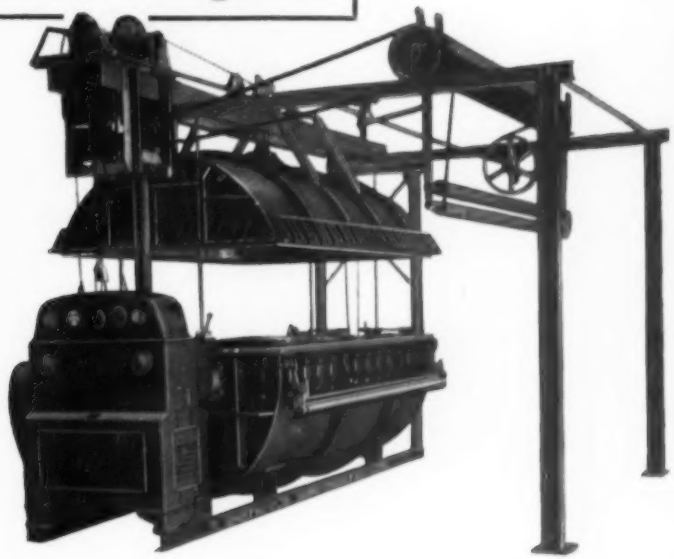

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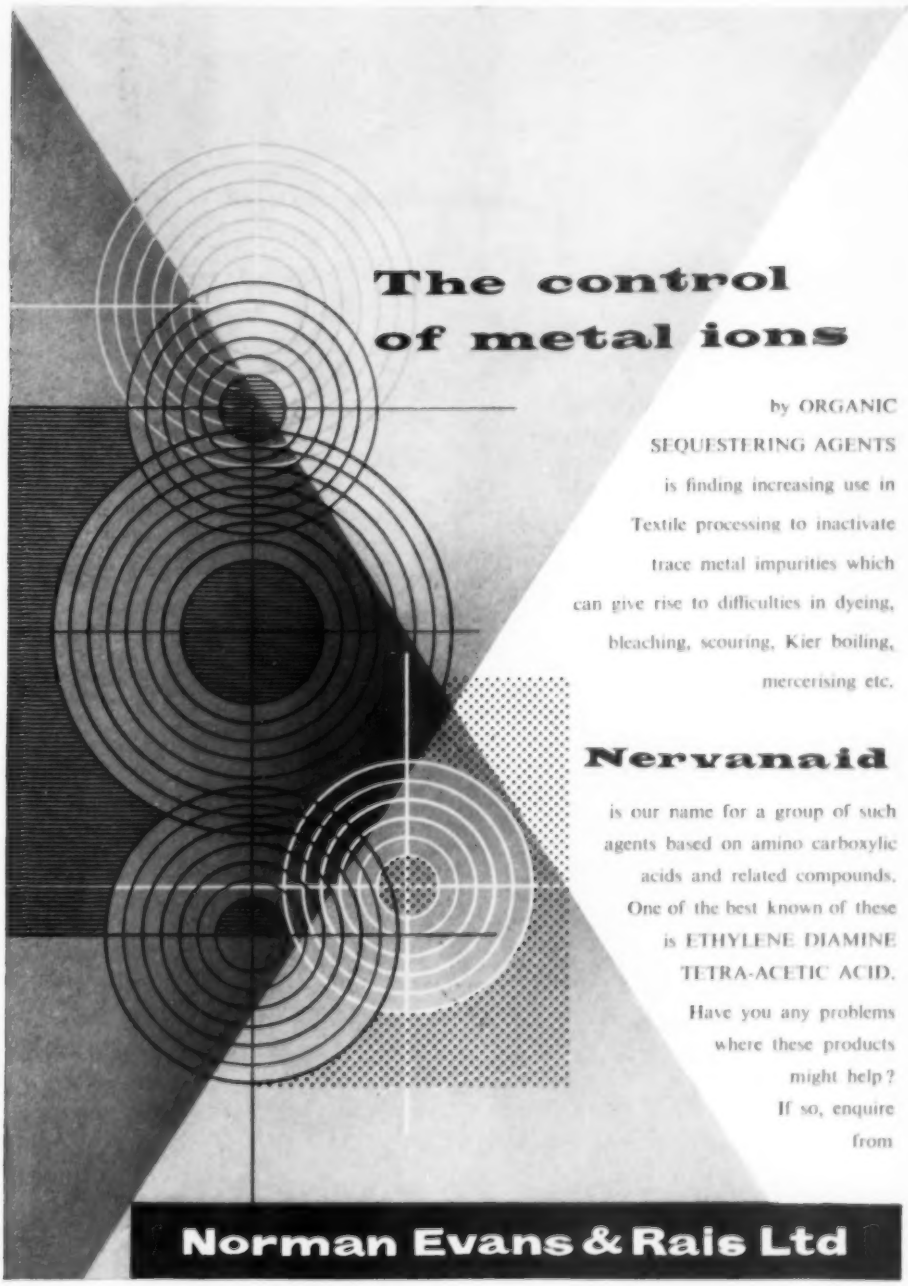

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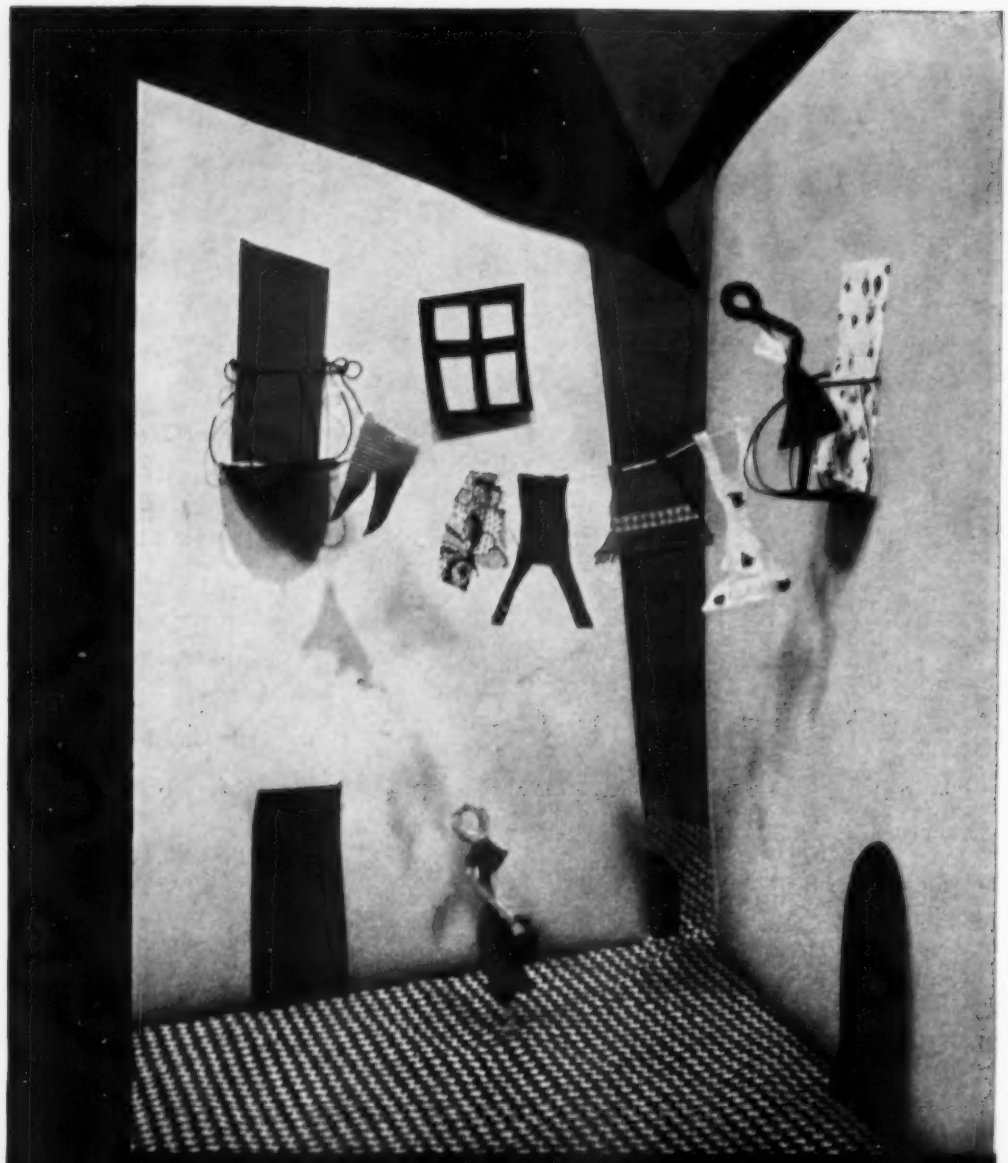

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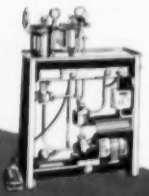

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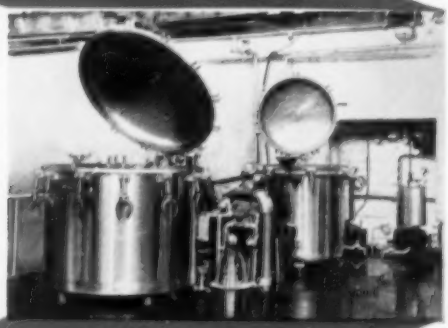

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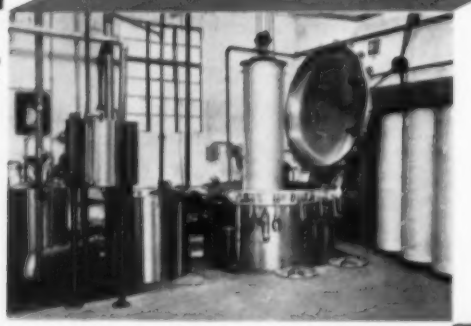

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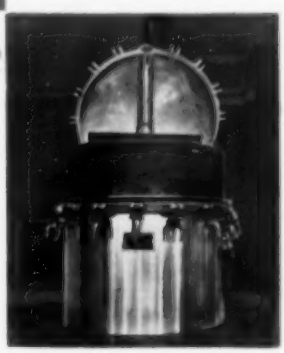

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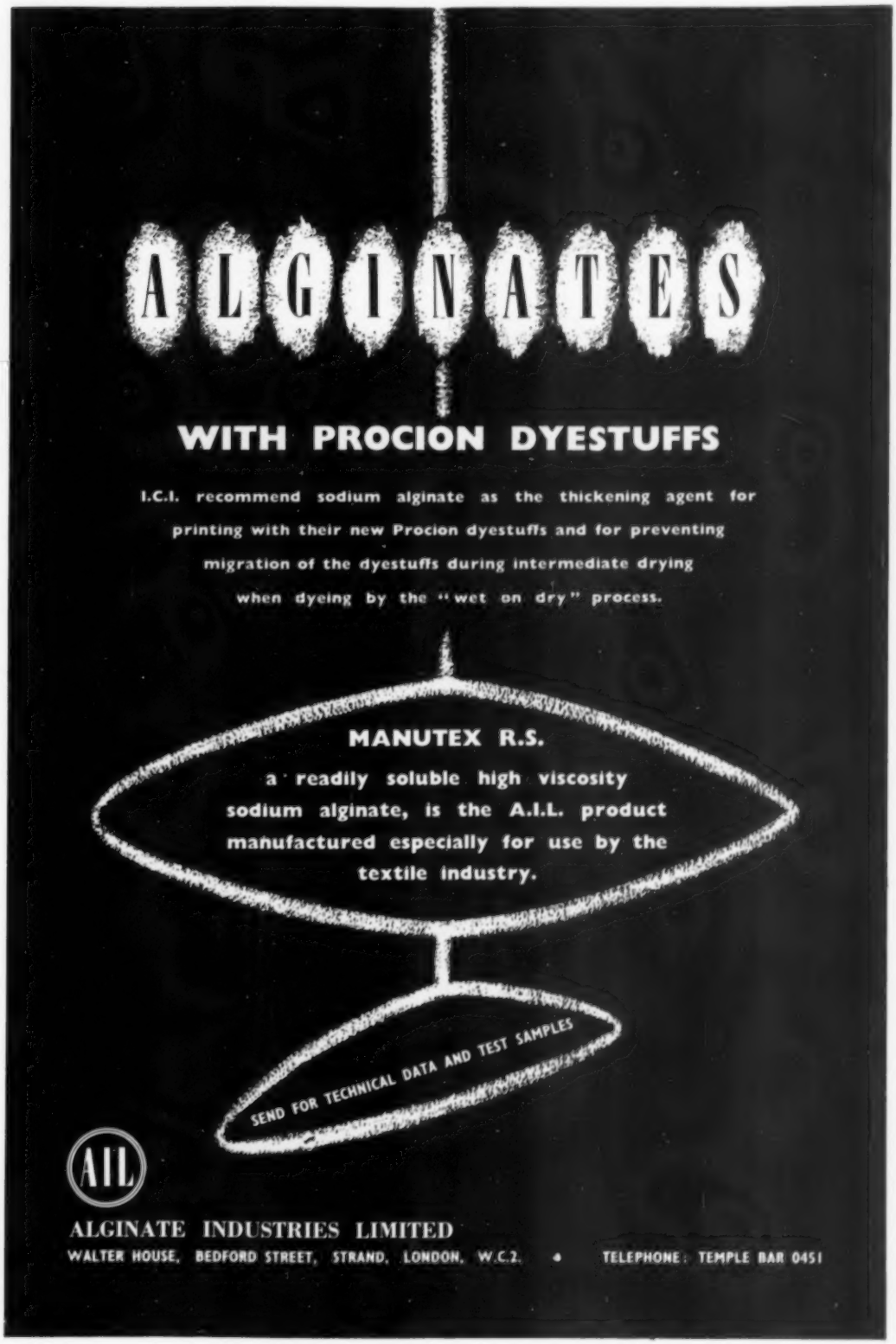
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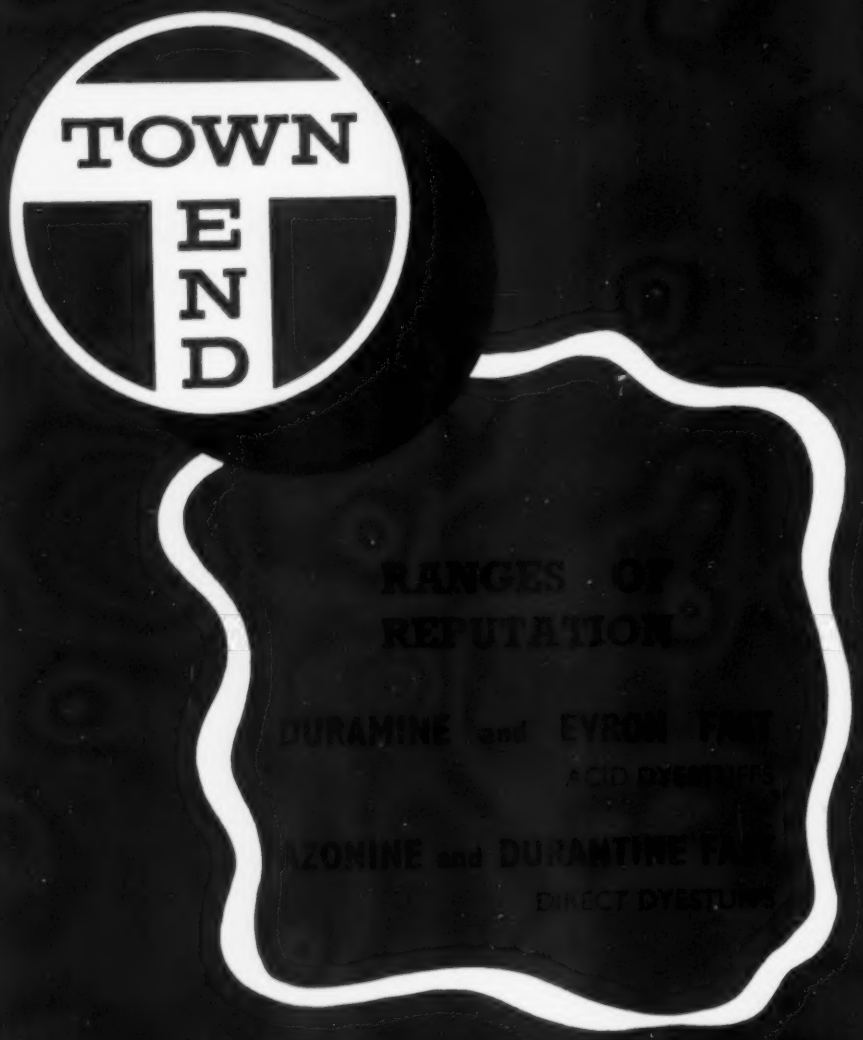

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Coating Plastic Material (XIII p. 443)

II—WATER AND EFFLUENTS

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III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

PATENTS

Trihydroxy Polyoxyalkylene Ethers of Glycerine—Emulsifying Agents
Dow Chemical Co. *BP* 777,978
BP 778,239

Trihydroxy polyoxyalkylene ethers of glycerol in which the glycerol nucleus is substituted by polyoxyalkylene ether chains each consisting of a polyoxy-1:2-propylene portion adjacent to the nucleus and a polyoxyethylene portion remote therefrom, are good non-ionic surface active agents. C.O.C.

Stable Anti-static Lubricants
ICI *BP* 770,415

An antistatic lubricant stable to the high temperatures of synthetic fibre processing, comprises an aqueous emulsion of a low viscosity mineral oil containing a mixture of different condensation products of ethylene oxide and a higher fatty alcohol, e.g. cetyl alcohol.
BP 770,416

The claims are restricted to application to polyester fibres. W.G.C.

Antistatic Agents
Boehme Fettchemie *BP* 775,976

An antistatic dressing for textile yarns comprises a mixture of an anion-active or non-ionic washing agent and the reaction product of equimolecular quantities of an anion-active compound and a cation-active onium compound other than phosphonium. W.G.C.

Yarn Lubrication
Ciba *BP* 773,574

For lubricating yarn during knitting a non-soapy anion-active lubricant, e.g. the sodium salt of phthalic acid monooctadecyl ether, is mixed with an evaporation retardant, e.g. terpineol, a dispersant, e.g. a polyglycol ether, a solution promoter, e.g. a monoether of ethylene glycol, a corrosion inhibitor, e.g. sodium nitrate, and a buffer, e.g. trisodium phosphate. Water is added to obtain the desired consistency. G.E.K.

Detergent Compositions
California Research Corpn. *BP* 777,523

Addition of a small proportion of a *para*-substituted straight-chain C_8–C_{18} acyl- or alkyl-phenol, e.g. *p*-tauroylphenol, to water-soluble organic sulphates and sulphonates greatly improves their foaming properties. C.O.C.

Detergent for use on Cotton
ICI *BP* 775,791

A mixture of a condensation product of 1 mol. of an alkyl phenol with 6–30 mol. of ethylene oxide, a water-soluble cellulose derivative, a *β*-monohydroxyethylamide of a fatty acid and an alkali, has good detergent power when used for cotton. C.O.C.

Stabilising Soap to Hard Water
Unilever *BP* 775,364

Addition of a water-soluble surface-active alkali metal salt of a compound of formula $R^1R^2N{\cdot}R^3{\cdot}COOM$ (R^1 = aliphatic hydrocarbon of 10–20 C; R^2 = H or aliphatic hydrocarbon of < 5 C; R^3 = hydrocarbon of 1–5 C chain length; M = Na or K), e.g. Na *N*-*n*-dodecyl-*γ*-amino butyrate, enables soap to be used in hard water without precipitation of insoluble lime soap. C.O.C.

Soapless Detergent Composition
South African Coal, Oil & Gas Corpn. *BP* 776,360

A mixture of aliphatic sulphonates of 15–19 C (75–95 parts by wt.) and aliphatic-sulphonates of 7–11 C (25–5) is not hygroscopic, has good detergent powers and does not irritate the skin. C.O.C.

Polyoxypropylene-Polyoxyalkylene Surface-active Agents
Wyandotte Chemical Corpn. *BP* 776,661

Modification of *BP* 731,603 (J.S.D.C., **71**, 470 (1955)).

Products having excellent detergent properties in cold water and ability to disperse lime soaps are obtained by condensing propylene oxide with a nitrogenous compound to form a polymer of mol. wt. ≤ 900 and in turn condensing that polymer with ethylene oxide. C.O.C.

Cold Water Soluble Products from Starch, Urea, and Formaldehyde
N.V. de W.A. Scholten's Chemische Fabriken
BP 772,479

Products useful as adhesives and sizes are obtained by heating a sufficiently disintegrated starch with a urea formaldehyde compound, e.g. dimethylol urea. W.G.C.

Derivatives of β-aminobutyric Acid
Heinkel *BP* 776,121

Derivatives of *β*-aminobutyric acid substituted on the N-atom are obtained by treating ethyl acetoacetate with an amine, and hydrogenating the product. These compounds are useful as coating agents for pigments, and as wetting and emulsifying agents. W.G.C.

Water-repellent Composition
Union Carbide & Carbon Corpn. *USP* 2,737,458

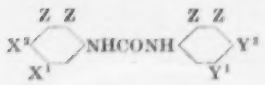
A composition for producing a non-durable water-repellent finish, i.e. one not fast to washing, and which can be obtained as a powder, flakes, etc., comprises active water repellents, emulsifiers, a boiling and insolubilising agent and a hygroscopic agent, e.g. paraffin wax (35% by wt.), carnauba wax (0·25), Al distearate (0·25), mineral spirits (3·5), sorbitan oleate (4·75), polyoxyethylene sorbitan oleate (4·75), water (20·65), polyvinyl alcohol (1·85), water soluble Al acetate (37% Al_2O_3) (18·5), sorbitol (10) and Na laurylsulphate (0·5). C.O.C.

Silicone Water-repellents
General Electric Co. *BP* 773,974

A water-repellent suitable for application to textiles or paper comprises (*a*) an organo polysiloxane containing no Si-bonded hydrogen and in which the organic groups are attached to Si by C–Si linkages, 2·5–15·0% of the organic groups being Alk of 8–12 C the remainder being CH_3, and (*b*) an incompletely condensed urea- or melamine-aldehyde condensate. Applied to cotton in the customary manner it yields a water-repellent finish which is resistant to numerous launderings or dry cleanings. C.O.C.

Diphenyl Urea Derivatives—Mothproofing Agents
Gy *BP* 774,802

Compounds of formula—



(X^1 = H or Hal when X^2 = CF_3 or *vice versa*; Y^1 = Hal, NO_2 or CF_3 when Y^2 = H, Hal or alkoxy *or vice versa*; Z = H or Hal), obtained by treating 2 mol. of a suitably substituted amino benzene compound with 1 mol. of a carbonic acid derivative, are insoluble in water but soluble in organic solvents. Dispersed in water, e.g. in a dyebath, they have affinity for wool to which they impart a mothproof finish which is fast to washing, milling, light and dry cleaning. Some of them have bactericidal and/or fungicidal properties. C.O.C.

Water-soluble Copper Compounds— Fungicides
FBy *BP* 777,544

Treating a copper salt of pentachlorophenol with excess of an organic nitrogenous compound yields excellent fungicides which give a colloidal solution in water and have good affinity for cellulose. The rotproof finish so given is extremely resistant to weathering and does not alter the colour of the fibres. Thus dry Cu pentachlorophenate (1 part by wt.) is heated at 45°c. with cetylbenzyldimethylammonium chloride (3) until a homogeneous water-soluble paste is formed. This paste when diluted with water is ready without further processing for treating cellulose fibres. C.O.C.

Compositions for Rendering Textiles and other Substrates Self-sterilising
Permachem Corpn. *BP* 777,679

A mixture of a sparingly water-soluble, white silver compound, e.g. silver chloride, and a light stabiliser for the silver compound is used. When a high degree of fungicidal effectiveness is also desired a sparingly soluble salt of a fungicidal organic acid is also included. This salt must be such that in contact with water it furnishes sufficient fungicidal anions but does not reduce the concentration of Ag ions below that necessary to exert the bactericidal effect of the Ag cation. Suitable salts are Al, Zn, Mg or alkaline earth metal salts of propionic, oleic, benzoic or salicylic acids and particularly Ca undecylenate. Starch, resins, etc., are suitable light stabilising agents. C.O.C.

Halogenated Alcohols as Solvents for Polyamides
British Nylon Spinners *BP* 775,382

Halogenated alcohols, e.g. 1:3-dichloropropanol-2, are excellent solvents for polyamides. C.O.C.

Coating Composition
FBy *BP* 776,087

A composition for coating any type of substrate but particularly films of poly(vinyl chloride), poly(vinyl acetate), cellulose acetate or nitrate or cloth impregnated with linseed oil, comprises an organic solvent solution of a poly*iso*cyanate-modified polyester containing free OH groups and a poly*iso*cyanate. After application it is cured by heating to ≯ 180°c. Thus the reaction product of 3 mol. adipic acid, 1 mol. trimethylolpropane and 3 mol. 1:3-butane diol (100 g.) are dissolved in *cyclo*hexanone (134) and butylacetate (66) and then treated with toluylene di*iso*cyanate (20·4) for 12 hr. at 100°c. until the viscosity was 3516 cp. 100 g. of the resulting solution was mixed with 10 g. of a 10% soln. of the reaction product of 1 mol. diethylethanolamine, 1 mol. phenyl*iso*cyanate and 15–30 g. of toluylened*iso*cyanate. Applied to poly(vinyl chloride) film (coating 10–30 g./sq.m.) and heat curing yielded a glossy uniform coating of very good adhesion to the poly(vinyl chloride). C.O.C.

Aqueous Emulsion of Poly Vinyl Acetate
British Oxygen Co. *BP* 775,038

Low viscosity highly stable emulsions of regular and uniform particle size are obtained by dispersing poly(vinyl acetate) in water in presence of a dispersing agent, a hydrophilic colloid as stabiliser, a peroxide or persalt as catalyst and a heavy metal salt capable of existing in more than one state of valency with a strong acid as the catalyst. Thus gum acacia (2·5), Na dioctyl sulphosuccinate (0·5) and $FeCl_3.6H_2O$ (0·002) were dissolved in water (42·1) and refluxed with vinyl acetate (54·0) and 30% H_2O_2 (0·7) for 2 hr. at 65–70°c. The temperature was then raised for 30 min. to 80°c. and the product cooled. Its viscosity at 20°c. was 100 cp. and the particle size 1·0–1·3 μ. The emulsions can be used for water based paints. C.O.C.

Water-dispersible Resinous Copolymers
Lewis Berger & Sons *BP* 774,868

Products which readily disperse in aqueous alkaline solutions and increase the viscosity of the medium to such an extent that gels may be formed at very low resin concentration are obtained by condensing a linear copolymer of an aromatic monovinyl compound and an $\alpha\beta$ ethylenically unsaturated dicarboxylic acid or anhydride with an alkyd resin. The linear copolymer should have viscosity ≮ 1000 cp. measured as a 30% soln. in methylethylketone and its compounds should be in the proportion of 1 : 1 to 4 : 1 in the order given. The proportions of copolymer to alkyd resin should be 1 : 3 to 3 : 1. The alkyd resin should have 10–60% excess hydroxyl over that necessary to esterify all its acidic components. The products are useful for coating paper, as thickeners in aqueous coating compositions and as a vehicle for carrying a light pigmentation in glazes used for overcoating surfaces in the application of designs. C.O.C.

Lowering the Viscosity of Emulsions of Polymers
Consortium für Elektrochemische Industrie *BP* 777,447

Addition of a small amount of H_2O_2 to emulsions of vinyl esters polymers or copolymers or styrene copolymers of high solid content and made by using polyvinyl alcohol or a water-soluble derivative thereof as the emulsifying agent, much reduces their viscosity. By this means finely dispersed emulsions of high solid content can be obtained. C.O.C.

Aminoplast Tanning Agents
L. Sellet *BP* 777,827

Treating an aminoplast resin having at least one reactive methylol group containing a reactive OH group, with a water-soluble amine base having at least one functional amino group, enough of this base being used to neutralise all the reactive methylol groups is used together with an anionic sulpho-containing water-soluble or water-dispersible compound as tanning agent. Thus melamine, HCHO and the sodium salt of disulphodinaphthylmethane are refluxed for 1 hr. at 80°c., urea added and refluxing continued for 4 hr. Chrome tanned calf skins treated with a solution of the product so obtained in an acid bath are given increased tensile strength, resistance to abrasion and increased flexibility. Similarly modified resins can be used for treating paper, textiles, etc. C.O.C.

Permanent Waving Composition
Ashe Laboratories *BP* 778,308

An aqueous paste containing a mercaptan and a normally solid non-ionic surface active agent is used, e.g. aq. ammonium thioglycollate (pH 9·2) and polyethylene glycol monostearate of mol. wt. about 400. C.O.C.

Cuprous Thiocyanate for After-treatment of Cellulose dyed or printed with Metallisable Direct Dyes (IX p. 440)

Standardisation of Test Methods for Textile Auxiliaries, II— Determination of Vat Acid Dispersion Power of Surface-active Agents (XIV p. 443)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Synthesis of *o*- and *p*-Benzenedisulphonic Acids
B. I. Karavaev and S. P. Starkov

J. Gen. Chem. U.S.S.R., **27**, 788–790 (March 1957)

Diazotisation of *o*- and *p*-aminobenzenesulphonic acids, replacement of the diazo group by the disulphide linkage, and subsequent oxidation give *o*- and *p*-benzenedisulphonic acids in 64% and 70% yield respectively. This method is found to be hazardous for aniline derivatives without the acid groups. G.J.K.

Reaction of Sulphonation. XLII— Preparation and Properties of Naphthalene-1:7-disulphonic acid and its Derivatives
A. A. Sprŷskov and O. S. Ivanova

J. Gen. Chem. U.S.S.R., **27**, 784–788 (March 1957)

The reaction of chlorosulphonic acid with Na naphthalene-2-sulphonate yields a mixture of 1:6- and 1:7-naphthalenedisulphonyl chlorides (I), which can be separated by crystallisation from organic solvents. Naphthalene-1:7-disulphonic acid (II) is prepared from 1-naphthylamine-7-sulphonic acid by diazotisation, replacement of the diazo group by disulphide, and subsequent oxidation. Solubilities of I in organic solvents and of the Na, Mg, Ca, and benzidine salts of II in water are investigated. G.J.K.

Nitration of 1-Ethylnaphthalene and of 4-Nitro- and 4:5-Dinitro-1-ethylnaphthalenes
S. I. Sergievskaya, T. S. Safonova, and G. Ya. Uretskaya
J. Gen. Chem. U.S.S.R., **27**, 749–754 (March 1957)
The nitration of 1-ethylnaphthalene and the chromatographic separation of the reaction products (whose structure has been proved) are investigated. G.J.K.

Fast Acid Dyes
K. Iwamoto
Yûki Gôsei Kagaku Kyôkaishi, **14**, 228–231 (1956): *Chem. Abs.*, **51**, 7017 (10 May 1957)
Review. C.O.C.

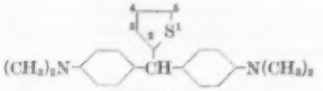
Chemical Constitution of Fast-to-light Direct Dyes
M. Sekido
Yûki Gôsei Kagaku Kyôkaishi, **14**, 228–231 (1956): *Chem. Abs.*, **51**, 7017 (10 May 1957)
Review. C.O.C.

Azoic Dyes
Y. Yamaguchi
Yûki Gôsei Kagaku Kyôkaishi, **14**, 239–245 (1956): *Chem. Abs.*, **51**, 7017 (10 May 1957)
Review, 37 references. C.O.C.

Disperse Dyes
K. Naiki
Yûki Gôsei Kagaku Kyôkaishi, **14**, 245–248 (1956): *Chem. Abs.*, **51**, 7017 (10 May 1957)
Review, 191 references. C.O.C.

Azo Dyes derived from o-Hydroxybenzeneboronic Acid Anhydride
H. Gilman, L. Santucci, D. R. Swayampati, and R. O. Ranck
J. Amer. Chem. Soc., **79**, 2898–2901 (5 June 1957)
Syntheses of the dyes 2-hydroxy-5-R-benzeneboronic anhydride are described (R = phenylazo, *p*-bromophenylazo, *o*-nitrophenylazo, *m*-nitrophenylazo, and *p*-nitrophenylazo). Better yields and/or quality of the unpurified dye were obtained if the time of coupling was prolonged. Ethylene chloride and nitrobenzene were both good solvents for purifying these dyes. The infrared spectra of the dyes have an absorption band at 9·1–9·2 μ which may be indicative of the C–B bond. C.O.C.

I— Triarylmethane Compounds as Redox Indicators in the Schoenemann Reaction. II— Syntheses of the Triarylmethanes
D. M. Smith, R. Blanchfield, J. L. Thompson, and G. A. Grant
Canadian J. Chem., **35**, 156–162 (Feb. 1957)
A series of substituted thiophen analogues of leuco Malachite Green (I) have been prepared by condensing the corresponding thiophenaldehyde with dimethylaniline by the use of anhydrous $ZnCl_2$. Instructions for preparing 16 thiophenaldehydes are given. The compounds (I) thus prepared were tested as redox indicators for the formation of perphosphonates by the action of alkali peroxide on *iso*propyl methylphosphonofluoridate. It is supposed that the perphosphonates oxidise chloride ions (from added HCl) to hypochlorite, which in turn oxidises the triarylmethane to the corresponding dye. The most efficient indicators (i.e. highest optical density of dye soln.) were 4:5-dialkyl derivatives, particularly when the 4-substituent is CH_3 and the 5-substituent has a longer carbon chain.



(I)

A.J.

Hydrolysis of Methyl and Sulpho Derivatives of Malachite Green
O. F. Ginzburg and P. M. Zavlin
J. Gen. Chem. U.S.S.R., **27**, 678–681 (March 1957)
Aminotriphenylcarbinols are prepared in good yield by adding a solution of Michler's ketone in dimethylaniline to the organic magnesium compounds. The effect of the methyl and the sulpho groups *ortho* to the central C on the hydrolysis constant of the triphenylmethane dye is investigated. In both instances the hydrolysis constant is appreciably lower compared with the unsubstituted triphenylmethane. G.J.K.

Polymethin Dyes
N. E. Podkletnov
J. Gen. Chem. U.S.S.R., **27**, 833–834 (March 1957)
A note on the paper "New Polymethin Dyes" by P. Pfeiffer and E. Enders (*Ber.*, **84**, 313 (1951)), claiming priority for the opening of the pyridine ring by acidic substances (Author's Licence No. 12098 (1945), U.S.S.R.). G.J.K.

Azamethin Dyes. II— Colour and Constitution of Acylacetamide Azamethin Dyes
G. H. Brown, J. Figueras, R. J. Gledhill, C. J. Kibler, F. C. McCrossen, S. M. Parmerter, P. W. Vittum, and A. Weissberger
J. Amer. Chem. Soc., **79**, 2919–2927 (5 June 1957)
Several *a*-acylacetamides have been synthesised and oxidatively condensed with 4-amino-3-methyl-*NN*-diethylaniline to yield azamethin dyes. The effects of substituents and of changes in solvent on the absorption spectra of these dyes are interpreted on the basis of electronic and steric factors and hydrogen-bonding. C.O.C.

"Threne" Vat Dyes
Y. Hosoda
Yûki Gôsei Kagaku Kyôkaishi, **14**, 234–239 (1956); *Chem. Abs.*, **51**, 7017 (10 May 1957)
Review, 5 references. C.O.C.

Polycyclic Aromatic Hydrocarbons. III— Substitution Derivatives of Anthanthrene
N. P. Buu-Hoï and D. Lavit
Rec. Trav. chim., **76**, 200–204 (Feb. 1957)
Anthanthrene reacts readily with *N*-methylformanilide and phosphorus oxychloride in the presence of *o*-dichlorobenzene to yield 6-formylanthanthrene. This, by reduction with hydrazine hydrate and KOH in diethylene glycol soln., gives 6-methylanthanthrene. In a similar way, formylation of the 6-methyl derivative gives 12-formyl-6-methylanthanthrene, reduction of which gives 6:12-dimethylanthanthrene. Anthanthrene reacts with sulphuryl chloride in *o*-dichlorobenzene to yield 6:12-dichloroanthanthrene. These reactions indicate the relative reactivity of the 6- and 12-positions in anthanthrene, corresponding to the *meso* positions in anthracene. A.J.

Reaction of Hydrazine with Nitroanthraquinones
N. S. Dokunikhin and I. D. Pletneva
J. Gen. Chem. U.S.S.R., **27**, 791–794 (March 1957)
The compound—

can be prepared from 1-nitroanthraquinone-2-carboxylic acid or the corresponding acid chloride by reaction with hydrazine. On the other hand, 1-amino-4-nitroanthraquinone-2-carboxylic acid is reduced by hydrazine to the corresponding 1:4-diamino derivative. G.J.K.

Fluorescent Brightening Agents
M. Okazaki
Yûki Gôsei Kagaku Kyôkaishi, **14**, 248–251 (1956); *Chem. Abs.*, **51**, 7017 (10 May 1957)
Review, 19 references. C.O.C.

Organic Phosphorescence
II— Revision of Lewis's Mechanism
S. Kato and M. Koizumi
Bull. Chem. Soc. Japan, **30**, 27–33 (Jan. 1957)
The relation between log I_p and time (where I_p = intensity of phosphorescence) for a trypaflavine–silica gel phosphor shows a marked deviation from linearity in the early stages. The decay is better represented by the expression—

$$I_p = Ae^{-\alpha t} + Be^{-\beta t} \qquad (i)$$

than by Lewis's expression $I_p = Ae^{-\alpha t}$ (A, B, α and β are constants). Variation in intensity of exciting light has no effect on the shape of the decay curve, nor has restriction of the spectral region over which the decay is measured. It is suggested that a metastable state other than, and in addition to, Lewis's triplet (T) state must exist. This state is designated the M state, and is supposed to be coupled with the T state, which is in turn coupled

with the singlet state. Transitions from the singlet to the ground state give phosphorescence; those from the T and M states are radiationless.

III— Quenching by some Gases and Vapours

S. Kato

Ibid., 34–37

The phosphorescence of the trypaflavine–silica gel phosphor is quenched by the following compounds in the vapour phase: O_2, NO, SO_2, styrene, ether, dioxan, *cyclo*-hexane, 2-ethyl-1-hexene, and methyl methacrylate; N_2, A, H_2, CH_4, *n*-C_6H_{14}, propylene, C_3H_8, acetone, CH_3OH, and H_2O have no effect. The quenched decay curves are still represented by equation (i) (above abstract), except at higher concn. of quencher, when the more general expression—

$$I_p = \Sigma a_i e^{-\alpha_i t}$$

should be used. From an analysis of the decay curves it is concluded that the triplet and metastable (M) states, but not the singlet state, are affected by the quencher, and also that triplet → singlet transition is not promoted.

IV— Quenching by Inorganic Salts

S. Kato, K. Kimura, and M. Koizumi

Ibid., 38–43

Phosphorescence of the trypaflavine–silica gel phosphor is unaffected by metallic ions, except Cu^{2+}, but the intensity of fluorescence is reduced. A.J.

Physical Measurement of Cone Pigment in the Living Human Eye

W. A. H. Rushton

Nature, **179**, 571–573 (16 March 1957)

Yellow light, too weak to bleach the pigments, is focussed upon the fovea through one half of the pupil, and the fraction reflected through the other half is measured by a photomultiplier. Brief illumination with a strong orange light causes bleaching of the pigment, and the rate of regeneration is then measured by measuring the intensity of the reflected yellow light. Regeneration is complete in ca. 6 min. The cone pigment cannot be rhodopsin (visual purple), which regenerates more slowly and is not bleached by orange light. Consideration of the displacements brought about by using bleaching lights of different colours points to there being two photolabile pigments in the cone, one of which may be iodopsin, previously isolated from the cones of chick eyes. Cone photosensitivity is much greater than that of rhodopsin, suggesting that light is brought to a focus in the cone and the pigment is concentrated at this focus. A.J.

Effect of some Copper Complexes on the Formation *in vitro* of Melanin

S. Isaka

Nature, 578–579 (16 March 1957)

The catalytic action of Cu^{2+} on the autoxidation of dihydroxyphenylalanine is enhanced by compounds which chelate with Cu^{2+}, e.g. pyridine, pyrrole, 2:2′-dipyridyl, cuproin, phenanthroline, and neocuproin. The catalytic activity varies with the mixing ratio of Cu^{2+} to chelating agent and is mostly max. when this is 1:2. The highest activity is shown by the 1:2 Cu^{2+}–neocuproin complex, and comparison with the phenanthroline complex suggests that the activity may be due to electron-releasing CH_3 groups in the 2- and 9-positions in neocuproin (2:9-dimethyl-1:10-phenanthroline). Cu^{+} complexes can be extracted from the reaction soln. A.J.

Organic Pigments

Y. Bansho and M. Fijitsuma

Yûki Gôsei Kagaku Kyôkaishi, **14**, 251–256 (1956); *Chem. Abs.*, **51**, 7017 (10 May 1957)

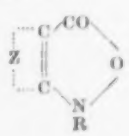
Review, 9 references. C.O.C.

PATENTS

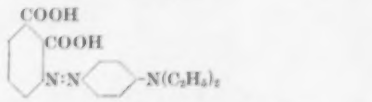
Colour Couplers

ICI *BP* 778,089

Compounds of formula—



(Z = atoms to complete a homo-, hetero- or polycyclic nucleus; R = H or acyl) are used as photographic colour couplers, e.g. 1-phenylcarbamyl-4-carboxy-$\beta\gamma$-benziso-oxazolone used as a colour coupler when developed with *p*-diethylaminoaniline yields the dye—

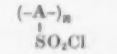


Their use results in images of better chemical stability and fastness to light than those given by azamethin dyes. C.O.C.

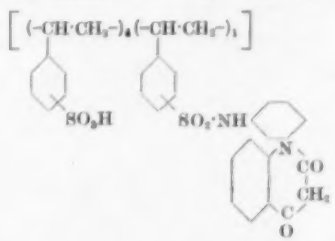
Non-diffusing Dye-forming Compounds for Use in Colour Photography

B. Gaspar *BP* 776,043

Compounds of formula HNR^1R^2, H_2NR^2 or HOR^1 (R^1 = radical causing the compound to act as a dye forming compound; R^2 = H, Alk or Ar; R^3 = aromatic radical having an available coupling position) are rendered non-diffusing by reacting them with compounds of formula—



(A = organic radical; $n = > 2$). Thus the compound

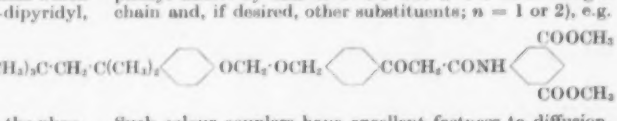


is obtained by condensing *N*-(*m*-aminophenyl)-1:2:3:4-tetrahydro-2:4-dioxoquinoline with the product obtained by the sulphur chlorination of Dow Resin PS–1 (a polystyrene of mol. wt. 6000). Coupled with diazotised 2-nitroaniline-4-sulphonic acid it yields a yellow dye. C.O.C.

Colour Couplers

Gevaert Photoprodukten *BP* 774,421

A coloured image is produced by developing with a primary aromatic amino developer, a reducible salt or a photographic element containing a colour coupler substituted by a group of formula $R{\cdot}O{\cdot}(CH_2{\cdot}CH_2{\cdot}O{-})_n$ (R = phenyl subst. by Alk of > 4 C but > 5 C in a straight chain and, if desired, other substituents; n = 1 or 2), e.g.

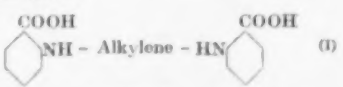


Such colour couplers have excellent fastness to diffusion. C.O.C.

Diazoamino Derivatives for use in Printing

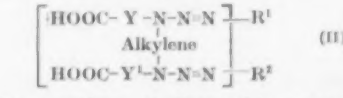
Fran *BP* 771,070

Diazoamino derivatives suitable for printing on textile fibres are obtained by reacting diazo-compounds in non-acid medium with—



(I)

(benzene nuclei may have Hal substituents; water-soluble salts of above compounds may be used) to give—



(II)

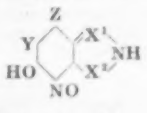
(R^1 and R^2 = residues of diazotisable mono-amine). Suitable diazo compounds include those derived from

aniline, toluidines, xylidines, cresidines, anisidines subst. or unsubst. by Hal, $-NO_2$, $-SO_2NH_2$ or $-CF_3$ groups. Examples of (I) are N:N′-dimethylene- and N:N′-hexamethylene-dianthranilic acid or their nuclear substituted halogeno derivatives prepared by condensing 1 mol. alkylenediamine with 2 mol. *o*-halogeno-benzoic acid in presence of Cu and a Cu salt. Examples of diazotisations and preparations of compounds (I) and (II) are given. The diazoamino compounds are mixed with coupling components, printed on textile fibres and developed by *neutral* steaming. The property of these diazoamino compounds of splitting rapidly by neutral steaming is especially valuable. E.T.

Nitroso Dyes

Fran *BP* 775,829

Wool and nylon are dyed in a weakly acid bath pure green to olive green of good general fastness by the ferrous complexes of nitroso-hydroxy-indazoles—



(X^1 = –CH or –C Hal when X^2 = N and vice versa; Y + Z = H or Hal). The dyes are prepared by nitrosating a hydroxy-indazole and then forming the ferrous complex, e.g. 6-hydroxy-indazole is dissolved in N-NaOH, $NaNO_2$ added and the mixture treated at 0–2°C. with N-H_2SO_4. The precipitated nitroso compound is stirred 16 hr. the temperature rising to atmospheric and is then dissolved by adding 10N-NaOH. The ferrous complex is prepared by adding aq. $FeSO_4$ filtering and drying at 90–95°C. The product dyes wool and nylon from a weakly acid bath. E.T.

Metal(Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

Ciba *BP* 775,011

The *o*-amino-*o*′-hydroxymonoazo dyes described in *BP* 757,485 (J.S.D.C., **72**, 603 (1956)) and *BP* 759,636 (J.S.D.C., **73**, 33 (Jan. 1957)) having a $CH_2{\cdot}SO_2$ substituent are made into asymmetric complexes by combining them with 1 atom of Co and 1 mol. of an *oo*′-dihydroxymonoazo dye, which preferably contains a sulphamyl group and is derived from an acetoacetanilide as coupling component. The products dye wool, nylon, etc. from weakly acid baths. Thus the monoazo compounds 5-nitro-2-aminophenol → 2-naphthylamine-6-methylsulphone and 2-aminophenol-4-sulphonmethylamide → *o*-chloroacetoacetanilide are dissolved together in aq. NaOH, and stirred at 80°C. with $CoSO_4$. The complex so formed dyes wool green. E.S.

Metal-complex Monoazo Dyes for Wool, Nylon, etc., having Sulphon-*N*-Alkyl-*N*-Hydroxyalkylamide Groups

Gy *BP* 775,005

The 1:2 Cr or Co complexes of suitable *oo*′-dihydroxy- or *o*-carboxy-*o*′-hydroxy-monoazo compounds, free from sulpho groups but containing sulphon-*N*-alkyl-*N*-hydroxyalkylamide groups, dye wool, nylon, etc., from neutral or weakly acid baths. They exhaust more completely from neutral baths than similar dyes having an unsubstituted $SO_2{\cdot}NH_2$ group. Thus 2-aminophenol-4-sulphon-*N*-methyl-*N*-hydroxyethylsulphonamide is diazotised and coupled with 1-*m*-chlorophenyl-3-methyl-5-pyrazolone in presence of soda ash. Boiling with aq. ammonium chromosalicylate gives the Cr complex which dyes wool orange. E.S.

Maroon Metal(Chromium)-complex Monoazo Pigment

ICI *BP* 776,873

The Mn salt of the 1:2 Cr complex of the monoazo compound 4-chloroanthranilic acid → 3:2-hydroxynaphthoic acid is a maroon pigment of good stability in nitrocellulose and similar lacquers, even in presence of metallic, e.g. Al, powders. E.S.

Azo Dyes Derived from Dextran

The Commonwealth Engineering Co. of Ohio *USP* 2,733,236

Dextrans produced from sucrose by fermentation are coupled with diazo compounds to give azo dyes, particularly suitable for wood-staining. Or a nitrobenzoic ester of a dextran is reduced with Na_2S to give an amino compound which may be diazotised and coupled with suitable coupling compounds. The *p*-aminobenzoic ester of a dextran of average mol. wt. 70,000 to 85,000, for instance, is diazotised and coupled with *β*-naphthol to give an orange. E.S.

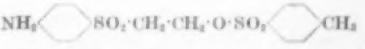
Monoazo Acid Dyes

Ciba *BP* 774,819

Monoazo compounds containing a group imparting solubility in water and also a group—

$$-SO_2{\cdot}(NH)_{n-1}(CH_2)_m{\cdot}O{\cdot}SO_2{\cdot}Z$$

(n = 1 or 2; m = 2 or 3; Z = Alk or aryl) dye wool from neutral or weakly acid baths. Thus, 4-(*β*-hydroxyethyl) acetanilide is converted into its *p*-toluenesulphonyl ester, and the acetyl group hydrolysed off to give the amino compound—

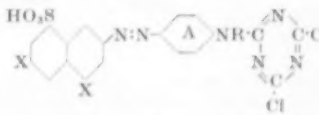


This is diazotised and coupled with 3-methyl-1-*p*-sulphophenyl-5-pyrazolone, to give a yellow monoazo acid dye. E.S.

Monoazo Dyes having a Cyanuric Ring, for Fixation on Cellulose by Alkaline Aftertreatment

ICI *BP* 774,925

Monoazo dyes—



(A may contain substituents other than OH and NH_2; R = H or Alk; one X = H, the other X = SO_3H) are applicable to cellulose by a process involving an aftertreatment in alkali + salt to give yellows and reds of good fastness to repeated washing. If R = Alk they have good fastness to spotting with alkali. Thus the monoazo compound 2-naphthylamine-4:8-disulphonic acid → *m*-toluidine, dissolved in water as its disodium salt, is treated with a soln. of cyanuric chloride in acetone, adding 2N Na_2CO_3 as acid binder. The product is padded on to cotton, dried at 20–40°C., and then immersed for 1 min. at 95°C. in 3% aq. NaOH containing 20% NaCl, to give reddish-yellow. E.S.

Bordeaux, Metal(Copper)-complex Disazo Direct Dyes

S *BP* 775,048

Urea di-J acid (I) is coupled with 1 mol. of a diazo compound derived from an *o*-aminophenol (or *o*-anisidine) having an alkyl- or phenyl-sulphonyl substituent, and with 1 mol. of a diazotised aniline having an *ortho* substituent capable of chelation and also optionally solubilising groups like SO_3H, $SO_2{\cdot}NH_2$, $SO_2{\cdot}NH$ alk. The disazo compounds so formed are converted into their copper complexes, which dye cellulose bordeaux. Thus coupling I first with 1 mol. of diazotised 2-aminophenol-4-sulphonamide and then with 1 mol. of 2-amino-4-methylsulphonylphenol, and heating at 80–85°C. with aq. $CuSO_4$ gives—

E.S.

Green Trisazo Direct Dyes for Aftercoppering

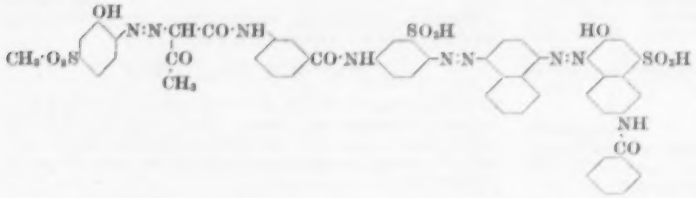
Gy *BP* 774,682

Trisazo dyes of the type—

$$A \rightarrow \text{acyl-CH{\cdot}CO{\cdot}NH-B-NH}_2 \rightarrow D \rightarrow E$$

(A = aniline deriv. having a metallisable group *ortho* to the NH_2 group; B = the residue of a monosulphonated substantive diamine in which one of the NH_2 groups is acylacetylated; D = 2-alkoxy-1-naphthylamine; E = *N*-acyl-*γ*-acid) give fast olive- to yellow-greens on cellulose by the aftercoppering method. Thus 2-amino-5-methylsulphonylphenol is diazotised and coupled with 4-(*m*-

acetoacetylaminobenzoylamino)aniline-2-sulphonic acid in presence of $NaHCO_3$. The product is salted out, filtered off, and diazotised and coupled with 1-amino-2-naphthoxyacetic acid in presence of acetic acid. The aminodisazo compound so formed is diazotised and coupled with *N*-benzoyl-γ-acid in presence of soda ash and pyridine, giving—

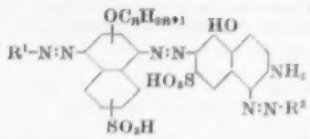


which dyes cellulose vivid yellowish-green, rendered fast to light, washing, and acid by aftercoppering. E.S.

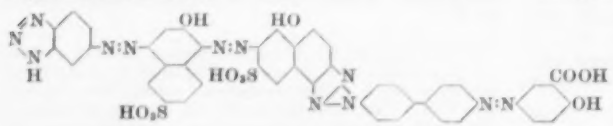
Trisazo Direct Dyes Having a Triazole Ring

Ciba *BP* 774,916

Tetrakisazo dyes—



(R^1 = salicylic acid residue, or a heterocyclic residue of > 1 hetero-atom at least one of which is N; R^2 = residue containing an azo link and an *o*-hydroxycarboxy group; n = 1 or 2) are treated with Cu compounds to give the metal complex, at the same time splitting off the group C_nH_{2n+1}, and converting the *o*-aminoazo system into a triazole ring. The chelated Cu is then removed to give mainly olive green direct dyes, which may be coppered in the dyebath or on the fibre. Thus the aminomonoazo compound 6-aminobenztriazole → 2-methoxy-1-naphthylamine-6-sulphonic acid is diazotised and coupled with the disazo compound salicylic acid ← benzidine $\xrightarrow{acid}$ J acid. The tetrakisazo compound so formed is heated at 90–95°C. for 4 hr. with ammoniacal $CuSO_4$, followed by de-coppering in 30% HCl in the cold, which gives the olive-green after-copperable trisazo dye—



E.S.

Production and/or Stabilisation of Hydroquinones

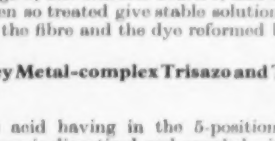
Gillette Industries *BP* 773,794

A quinone or hydroquinone when treated with an aqueous alkaline solution of an alkali metal borohydride yields a stabilised solution. Quinonoid dyes, e.g. C.I. Natural Orange 6, C.I. Natural Black 7, and C.I. Mordant Black 37, when so treated give stable solutions which can be applied to the fibre and the dye reformed by oxidation *in situ*. C.O.C.

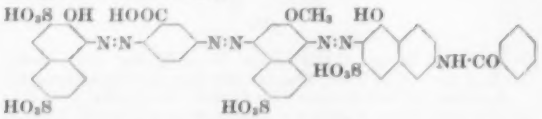
Blue and Grey Metal-complex Trisazo and Tetrakisazo Direct Dyes

ICI *BP* 755,450

Anthranilic acid having in the 5-position a nitro or acylamino group is diazotised and coupled with a suitable naphtholsulphonic acid, and the nitro or acylamino group is then converted to NH_2, to give an aminoazo compound—



(*x* = H, or an alkylated, *cyclo*alkylated, aralkylated, arylated, or acylated amino group; *m* = 1, 2, or 3; the OH group is *ortho* to the azo link). This is then diazotised and coupled with an *o*-alkoxy-aniline or -naphthylamine, and the aminodisazo compound so formed is then diazotised and coupled with a suitable naphthol, which may carry a substituent *N*:*N*-pyrazolone or *N*:*N*-aryl group. The tris- or tetrakisazo compounds so formed (which may also be made by alternative syntheses) may be made into Cu or Ni complexes, and in the metal-free or metal-complex form dye cellulose blue and grey whose light fastness is not appreciably decreased by application of a crease-resistant resin. Thus the monoazo compound 5-nitroanthranilic acid → R acid is reduced with Na_2S to give the amino compound, which is diazotised and coupled with 2-methoxy-1-naphthylamine-6-sulphonic acid. The aminodisazo compound so formed is diazotised and coupled with *N*-benzoyl-J acid in presence of pyridine bases to give—



Boiling with ammoniacal $CuSO_4$ gives the copper complex which dyes cellulose blue-grey. E.S.

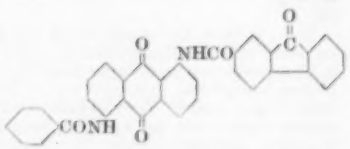
Molecular Mixtures of 1:5- and/or 1:4-Diacylaminoanthraquinone Vat Dyes

Ciba *BP* 775,885

Unexpected and valuable dyeing properties are shown by molecular mixtures of 1:5- and/or 1:4-diacylaminoanthraquinone vat dyes. It appears that molecular compounds may be formed from the two components of the mixture, e.g. by dissolving the mixed powders in H_2SO_4 and reprecipitating by the addition of water *or* by applying the mixed dyes in the usual reduced form to the cellulosic fibres, oxidising and then treating the dyeings with boiling 0·3% soap soln. for 30 min. The dyeing is generally at least as pure as that given by the component normally giving the purer tint and strong bathochromic action occurs in some cases. Thus equimolecular proportions of 1:4-dibenzoylaminoanthraquinone (dull red-orange) and 1-(4′-dimethylsulphonamidobenzoylamino)-4-benzoylaminoanthraquinone (bright red) when applied by vatting, oxidation and soaping at the boil give beautiful violet dyeings of good fastness. E.T.

Anthraquinone Vat Dye

Cassella *BP* 777,789

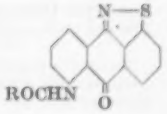


is produced by condensing fluorenone-2-carboxylic acid or its functional derivatives with 1-amino-5-benzoylaminoanthraquinone. Thus, *o*-dichlorobenzene (350), thionyl-chloride (24) and fluorenone-2-carboxylic acid (16·8) are heated 4 hr. at 160°C. Excess thionyl chloride is removed by vacuum distillation and 1-amino-5-benzoylaminoanthraquinone (17) is added. After 12 hr. at 160°C., the melt is cooled and filtered. The product is washed with *o*-dichlorobenzene and then with methanol before drying. The resulting vat dye gives bright golden-orange dyeings and prints of outstanding fastness on vegetable fibres. E.T.

***iso*Thiazolcanthrone Dyes**

BASF *BP* 775,957

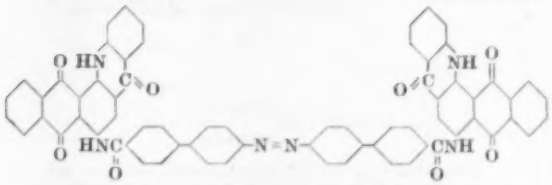
Yellow dyes for wool, silk and polyamide fibres of excellent fastness to light and washing have the formula—



(R = mono-sulphonated Alk of 1–5 C). They are produced by reacting a 5-acylamino-1:9-*iso*thiazoleanthrone, in which the acyl group is the residue of a halogenated fatty acid, with an alkali metal sulphite. Thus, 5-(2′-chloro)-propionylamino-1:9-*iso*thiazoleanthrone (5), 40% $NaHSO_3$ (65), and 30% NaOH (34) are heated in an autoclave 7 hr. at 130°c. Reaction mass is poured into water (1000), heated to 95°c. and filtered. The hot filtrate is treated with Na_2SO_4 (100) and the product precipitated on cooling is filtered, washed with aq. 10% Na_2SO_4 and dried. E.T.

Anthraquinone-Acridone Vat Dye

General Aniline *USP* 2,729,631

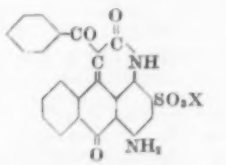


is produced by condensing 4-amino-2:1-benzacridone (0·2 mol.) with azodiphenyl-4:4′-dicarbonylchloride (0·1 mol.) in pyridine and dichlorobenzene at 110–115°c. The condensation melt is finally cooled to 25°c., filtered and the insoluble product washed with alcohol and dried. The dye obtained gives unique yellowish-green dyeings of good fastness to chlorine. E.T.

Anthrapyridone Dye

BASF *BP* 776,513

The scarlet red dye of formula—

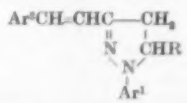


(X = H or alkali metal) gives dyeings of excellent fastness to light and washing on wool, silk and polyamide fibres. It is especially suitable for use with polyacrilonitrile fibres. It is made by treating 3′-benzoyl-2-halogeno-4-amino-1:9-anthrapyridone in aqueous medium with an alkali metal sulphite at 100–160°c. for 10–24 hr. in an autoclave. Alternatively, 3′-benzoyl-4-(4′-methyl)-phenylsulphonyl-amino-1:9-anthrapyridone is hydrolysed by 90–100% H_2SO_4 at 20–40°c. to give the same final product. E.T.

Fluorescent Pyrazoline Dyes for Use in Dyeing and in Coatings for Fibrous Materials

Ciba *BP* 771,311

Water-soluble or insoluble fluorescent dyes—

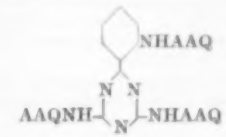


(Ar^1 and Ar^2 = subst. or unsubst. aromatic residues; R = H or hydrocarbon radical) made by condensing ketones ArCH = CHCOCH = CHR with arylhydrazines Ar^2NHNH_2 are applied to fibrous materials by addition to coating compositions. Thus, a solution of the Na salt of a sulphonated 1:5-diphenyl-3-styrylpyrazoline (0·4) in water (5) is added with stirring to a 75% solution in butanol of a customary HCHO–urea condensation product etherified with butanol (40). The coloured resin solution is condensed 3–4 hr. at 135–40°c. and the hardened resin then ground to form a pigment. The pigment (100) is suspended in an alcoholic polyvinyl acetate lacquer (1000) and applied to paper, cardboard or fabric by spray-gun. The coating is air-dried. The dyes may also be applied by dyeing from aqueous or solvent solutions on nitrogenous fibres. Those with strong colouring power may be used alone but weaker ones can be used with other dyes to which they impart enhanced brilliance. E.T.

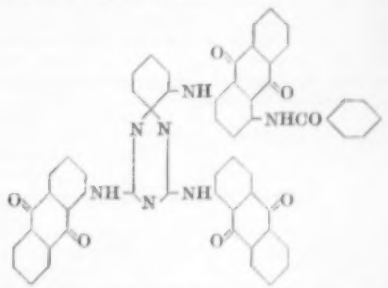
Phthaloylcarbazolotriazine Vat Dyes

Cyanamid *USP* 2,729,638

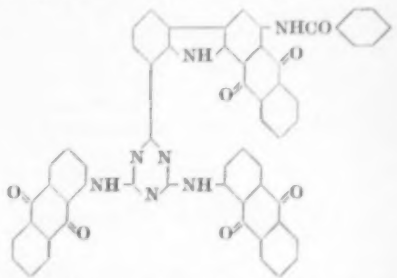
Brown to bordeaux to olive-green vat dyes of high fastness are produced via intermediates of formula—



(AAQ = *a*-anthraquinonyl radical). In one example this type of intermediate is made by adding 2-(*o*-chlorophenyl)-4:6-di-(*a*-anthraquinonyl)-1:3:5-triazine (12·7), 1-amino-4-benzamidoanthraquinone (6·9), Na_2CO_3 (5), anhydr. Na acetate (3) and cuprous bromide (0·5) to molten naphthalene (100) at 100°c. and heating until reaction is complete at 205°c. The melt is then cooled to 150°c., diluted with chlorobenzene (185) and cooled to 100°c. After filtration the remaining solvents are removed by steam distillation and the product is further purified by HCl extraction. The intermediate product (6)—



is then added to $AlCl_3$ (12) and nitrobenzene (40), fused at 50°c. and the melt is gradually heated to 75°c. The mixture is drowned into excess dil. NaOH; the nitrobenzene removed by steam distillation and the product—



isolated by filtration and washing. These vat dyes are unusual in that they give brown to green dyeings instead of the yellows to reds usually obtained with triazine vat dyes. E.T.

Stilbyl Triazole Fluorescent Brightening Agents

Gy *BP* 774,010

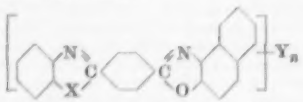
2-(Stilbyl-4″)-(4:5 benzo or naphtho)-1:2:3-triazoles containing in the stilbene radical in at least one *o*-position or in the *p*-positions to the ethylene bridge a sulphonyl substituent which does not dissociate acid in neutral water, have vivid blue to green-blue fluorescence in organic solution. They are used in lubricating oils, paraffin wax, cosmetics, synthetic polymers, etc. Thus the sodium salt of 2-(stilbyl-4″)-(naphtho-1′:2′:4:5)-1:2:3-triazole-2″-sulphonic acid is slowly added to a mixture of PCl_5 and $POCl_3$ and heated to 102–105°c. for 3 hr. It is then poured into a mixture of ice and water so that the temperature is < 5°c. when 2-(stilbyl-4″)-(naphtho-1′:2′:4:5)-1:2:3-triazole-2″-sulphonic acid chloride separates out. This is dissolved in dry nitrobenzene. To this solution phenol is added at 55–60°c. and then aqueous NaOH slowly added and the whole heated for 3 hr. at 90–95°c. The nitrobenzene and excess are then steam

distilled off and the residue cooled, well washed with water and recrystallised from acetic acid. It consists of 2 - (stilbyl - 4″) - (naphtho - 1:2′:4:5) - 1:2:3 - triazole - 2″ - sulphonic acid phenyl ester. It dissolves in many organic solvents to yield strongly fluorescent solutions. It is used for brightening rubber, oil, waxes and in particular, synthetic substances such as vinyl and vinylidene polymers, polystyrene, polyethylene, etc. C.O.C.

2-[*p*-(2-Benzazoyl)phenyl]-naphth[1,2]oxazoles and their Monosulpho Derivatives—Fluorescent Brightening Agents

DuP *USP* 2,737,516

Compounds of formula—

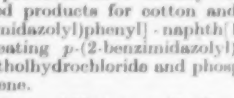


(X = O, S or NH; Y = sulpho; *n* = 0 or 1) are fluorescent brightening agents of good fastness to chlorine. The unsulphonated products have affinity for cotton, the monosulphonated products for cotton and nylon. Thus 2 - [*p* - (2 - benzimidazolyl)phenyl] - naphth[1,2]oxazole is obtained by heating *p*-(2-benzimidazolyl)-benzoic acid, 1-amino-2-naphtholhydrochloride and phosphoryl chloride in trichlorobenzene. C.O.C.

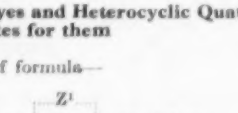
Polymethin Dyes and Heterocyclic Quaternary Salts as Intermediates for them

Kodak *BP* 774,780

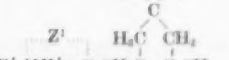
Compounds of formula—



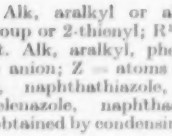
or



(R^1 = Alk or subst. Alk, aralkyl or alkenyl; R^2 = carbocyclic aromatic group or 2-thienyl; R^3 = H or CH_3; R^4 = H, Alk or subst. Alk, aralkyl, phenyl or subst. phenyl, or furyl; X^1 = anion; Z = atoms to complete a thiazole, benzthiazole, naphthathiazole, benzoxazole, naphthoxazole, benzselenazole, naphthaselenazole or thiazoline nucleus) are obtained by condensing a compound of formula—



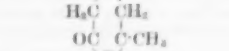
with one of formula—



Thus 3 - ethyl - 2 - (2 - phenyl)propenylbenzthiazolium - *p* - toluenesulphonate is obtained by condensing 3-ethyl-2-methylbenzthiazolium-*p*-toluenesulphonate with acetophenone. They are used as intermediates for polymethin dyes.

BP 774,779

Polymethin dyes are obtained by condensing an intermediate of the above type with a compound of formula—

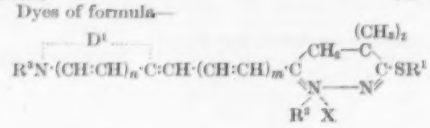


(R^5 = Alk or subst. Alk; R^6 = carboxylic acyl group; R^7 = phenyl or substituted phenyl; X^2 = anion; *d* = 1, 2 or 3; Z^2 = atoms to complete a 5- or 6-membered ring; *n* = 0 when Z^2 completes a 5-membered ring and 0 or 1 when Z^2 completes a 6-membered ring). Thus 3:3′-diethyl-11:13-neopentyleneoxathiatricarbocyanine iodide is obtained from 3 - ethyl - 2 - (1:5:5 - trimethyl - 3 - *cyclo* - hexenylidene)methylbenzthiazolium iodide, 2-β-acetanilidovinyl-3-ethylbenzoxazolium iodide and trimethylamine by heating them together in solution in pyridine. C.O.C.

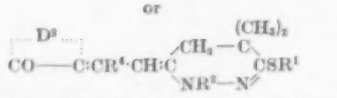
Cyanine Dyes

Ilford *BP* 775,386

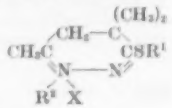
Dyes of formula—



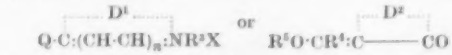
or



(R^1 = Alk; R^3 = Alk, hydroxyalkyl or aralkyl; R^4 = Alk or aralkyl; R^5 = H or Alk; *n* and *m* each = 0 or 1; X = acid radical; D^1 = residue of a 5- or 6-membered ring; D^2 = residue of a ketomethylene system) are obtained by condensing a compound of formula—



with one of formula—

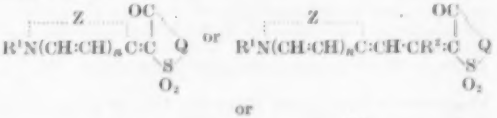


(Q = thioether, thioethervinyl or acetanilidovinyl; R^5 = Alk). Thus 4:4:6-trimethyl-3-methylthiopyridazinine, 2-methylthiobenzothiazole and methyl *p*-toluene sulphate are fused together for 1 hr. at 130° C. and then refluxed with pyridine for 1 hr. On pouring into 2% aq. KI the orange dye (1:4:4-trimethyl-3-methylthio-6-pyridazinine)(3-methyl-2-benzothiazole)monomethinecyanine iodide is precipitated. C.O.C.

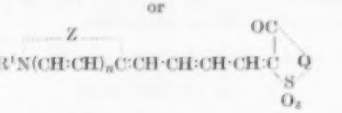
***mero*Cyanine Dyes**

Kodak *BP* 776,414

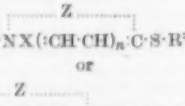
Dyes of formula—



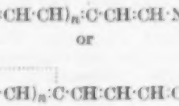
or



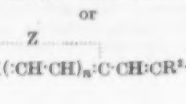
(R^1 = subst. or unsubst. Alk; Q = atoms to complete a 5-membered ring containing 3 C, 1 S and 1 N or C atom; Z = atoms to complete a 5- or 6-membered ring; *n* = 0 when Z completes a 5-membered ring and 0 or 1 when Z completes a 6-membered ring; R^2 = H or Alk) are obtained by condensing a compound of formula—



or

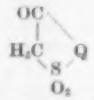


or



or

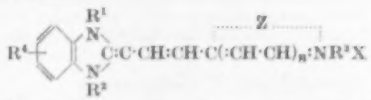
(R^2 = Alk or Ar; R^4 = aryl; R^5 = Ar; X = acid radical) with one of formula—



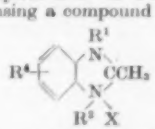
Thus 3-methyl-2-phenyl-4-thiazolidone-1:1-dioxide, 2-β-acetanilidovinyl-3-ethylbenzoxazolium iodide and triethylamine refluxed in ethyl alcohol for 30 min. yield the dye 5-[(3-ethyl-2-benzoxazolylidene)ethylidene]-3-methyl-2-phenyl-4-thiazolidone-1:1-dioxide. C.O.C.

Benzimidazolocarbocyanine Dyes
Kodak *BP* 776,050

Dyes of formula—



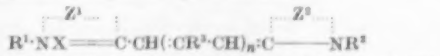
(R^1 = Alk, alkyl or Ar; R^2 and R^3 = Alk or alkyl; R^4 = acetyl, Alk·NHCO– or Br; X = acid radical; n = 0 or 1; Z = atoms to complete a 5- or 6-membered ring) are obtained by condensing a compound of formula—



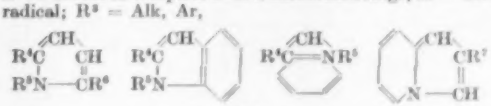
with chloralalcoholate. Thus 1:3-diethyl-5-ethylcarbamyl-2-methylbenzimidazolium iodide condensed with chloralalcoholate yields the red dye 1:1′:3:3′-tetramethyl-5:5′-di(ethylcarbamyl)benzimidazolocarbocyanine iodide. C.O.C.

Mixture of a Cyanine and a Benzimidazolocarbocyanine Dye as a Supersensitiser
Kodak *BP* 776,049

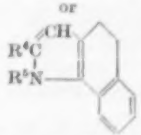
A mixture of a cyanine dye of formula—



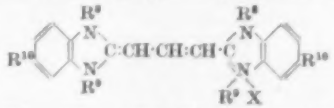
(R^1 and R^2 = subst. or unsubt. Alk; n = 0 or 1; Z^1 and Z^2 = atoms to complete 5- or 6-membered rings; X = acid radical; R^3 = Alk, Ar,



or



(R^4 and R^5 = H, Alk or Ar; R^6 = H or Alk; R^7 = Ar) and a benzimidazolocarbocyanine dye of formula—



(R^8 and R^9 = Alk or allyl; R^{10} = Br, acetyl or ethylcarbamyl) e.g. 1′-ethyl-3-methylthia-2′-cyanine iodide and a mixture of 5:5′-diacetyl-1:1′:3:3′-tetraethylbenzimidazolocarbocyanine iodide, is used as a photographic supersensitiser. C.O.C.

Furnace Black
Godfrey L. Cabot *BP* 773,777

An atomised conical spray of hydrocarbon oil is directed along a heat-insulated reaction chamber. The entrance end of the chamber is flared around the spray. A combustible gas and air are fed into a combustion space situated behind the spray. This space is formed by a confined extension of the reaction chamber so that the gases flow continuously along it toward the spray and the fuel gas is all burnt up before the spray is reached. The products of combustion intermingle with the spray causing the hydrocarbons to decompose and form furnace black. A high yield of high quality black is obtained. C.O.C.

Carbon Black
Godfrey L. Cabot *BP* 778,207

A method of increasing the yield of carbon black and synthesis gas from liquid hydrocarbons. C.O.C.

Carbon Black
International Carbon Black Co. *BP* 778,247

Compressed gaseous hydrocarbon and compressed air are separately fed into the apparatus, one of them into an antechamber whence it passes to the reaction chambers and the other directly to the reaction chambers. This gives uniform mixing of the gases without risk of explosion. C.O.C.

Carbon Black Pellets for Inks
Godfrey L. Cabot *BP* 774,944

A fluffy ink grade furnace black is treated with 2–15% of an oil which is neither subject to oxidation polymerisation nor readily evaporable. It is then subjected to turbulent agitation by any convenient means, e.g. pelletisers of the type described in *USP* 2,120,540 and 2,306,908, until pellets of the desired density are formed. The resulting pellets are readily dispersed in inks to yield a product well within grit and 325 mesh residue specifications. C.O.C.

Titanium Dioxide
DuP *USP* 2,737,460

The fineness and gloss characteristics of TiO_2 are improved by dry grinding it in presence of 0·005–0·5% triethanolamine and 0·05–5·0% of an aerogel capable of reducing metallic erosion during the grinding, e.g. a white silica aerogel. C.O.C.

Aqueous Dispersions of Pigments
L. Auer *BP* 774,079

Stable aqueous dispersions are produced by slurrying a hydrophobic organic pigment or carbon black (14–35 parts by wt.) with water (65–86) in presence of a fatty alcohol sulphate (9–40% on the dry wt. of the pigment). Treating press cake in this manner yields a highly concentrated dispersion which can be diluted for use, e.g. with a resin binder for textile printing. C.O.C.

Pelleting Carbon Black (I p. 428)
Reactive Dyes for Textiles (VIII p. 439)
Dyes for the Hydrophobic Fibres (VIII p. 439)
Oxidation Dyes on the Fibre (VIII p. 439)
Dyeing of Polyester Fibres with Disperse Dyes (VIII p. 439)
Iron Phthalocyanine as Peptising Agent for Rubber (XIII p. 442)
Stilbenebisazo-β-naphthol as Indicator in the Volumetric Determination of Sulphate Ions (XIV p. 443)
Chromatography of Polynitronaphthylamines (XIV p. 443)

V— PAINTS; ENAMELS; INKS

PATENTS

Oil and Water Emulsions
Anzin *BP* 774,792

Water and oil emulsions stabilised by solid particles are converted from water-in-oil to oil-in-water or *vice versa* in which the contact angle at the solid/oil/water interfaces is controlled from $< 90°$ to $> 90°$ or *vice versa* by coating the solid particles with a surface active agent having a hydrocarbon chain of appropriate length. Thus C.I. Pigment White 5 or 21 (2·5 g.), M/10,000 Na dodecyl sulphate (10 ml.) and benzene (10 ml) yield an oil-in-water emulsion. Increasing the strength of the Na dodecyl sulphate to M/100 inverts the system to a water-in-oil emulsion. C.O.C.

Coating Compositions
DuP *BP* 774,625

Modification of *BP* 654,446 (J.S.D.C., **67**, 391 (1951)). Limiting the water content of the composition to $> 1\%$ by weight enables the composition to be applied and cured to yield haze-resistant finishes irrespective of the atmospheric humidity prevailing at the time of application. C.O.C.

Cellulose Nitrate Dipping Lacquers
Wasag-Chemie *BP* 776,492

A lacquer which gels at $< 50°C$. consists of cellulose nitrate dissolved in a mixture of propanol and/or high

alcohol, and a benzene hydrocarbon and/or a chlorinated hydrocarbon. The components of the mixture are chosen so that they dissolve the cellulose nitrate at ≯ 70°C. C.O.C.

Coating with Metallic Silver
Olin Mathieson Chemical Corpn. *BP* 776,508
The surface to be coated is treated with an aqueous ammoniacal silvering solution containing β-hydroxyethylhydrazine as the reducing agent. Such a solution is particularly applicable by spraying. C.O.C.

Sheep Marking Fluid
Lord Mayor, Aldermen and Citizens of the City of Bradford *BP* 776,446
A water-in-oil emulsion for sheep marking consists of water emulsified in a mixture of a volatile hydrocarbon spirit solvent, *e.g.* coal tar naphtha, a pigment, e.g. C.I. Pigment Red 3 or C.I. Pigment Blue 15, and City of Bradford Recovered Wool Grease. The latter component may be replaced by a mixture consisting predominantly of City of Bradford Hand Neutralised Wool Grease and City of Bradford Wool Grease Fatty Acids. They can be applied to wet sheep without running or blurring, weather well and are completely removed when the wool is washed in the normal manner. C.O.C.

Water-dispersible Resinous Copolymers (III p. 430)

VI—FIBRES; YARNS; FABRICS

Influence of Pretreatment on the Reactivity of Cotton as measured by Acetylation
R. J. Demint and C. L. Hoffpauir
Text. Research J., **27**, 290–294 (April 1957)
The reactivity of cotton as measured by perchloric-acid-catalysed acetylation depends on whether the cotton is in the form of fabric, yarn, or sliver. It is suggested that the differences in reactivity are due to greater retention of the heat of reaction within the structure of the yarn or fabric, resulting in a higher reaction temperature. Pretreatment with dilute sodium hydroxide solution, decrystallisation, and extraction with alcohol and with water all increase the reactivity of cotton. When decrystallisation is followed by solvent exchanging the moisture with acetic acid without drying, the rate of acetylation is further increased. There are indications that solvent exchange would also supplement the increased reactivity brought about by other pretreatments. Boiling in water for a short time is a simple and effective method of increasing the reactivity of cotton. J.C.F.

Alginate Hessian
L. A. Bashford, M. D. Eastham, J. P. Hilton, W. S. Holden, L. Horton, R. S. Thomas, and F. N. Woodward
J.S.D.C., **73**, 203–208 (May 1957)
Alginate hessian suitable for military camouflage use has been fabricated from pigmented continuous-filament yarn using the interlace knitting technique. Resistance to use and weathering was enhanced by chromating the fabric thus obtained and coating with pigmented, chlorinated paraffin wax emulsified in sodium caseinate solution. AUTHORS

PATENTS

Filaments, Films, etc. from Acrylonitrile Polymers
Chemstrand Corpn. *BP* 775,892
Products less brittle and of increased extensibility and thermal stability are obtained if after extrusion and congulation they are stretched for 15 sec. to 30 min. at 100–80°C. to orient the molecules, relaxed and then crystallised by treating at 110–120°C. for 15 sec. C.O.C.

Polyacrylonitrile Films or Filaments of Improved Dyeing Properties
Courtaulds *BP* 775,887
A polyacrylonitrile solution containing a condensate of epichlorhydrin and a primary or di-secondary amine, e.g. butylamine or piperazine, is extruded into a coagulating bath and the resultant film or filament while still wet and swollen treated with a solution of a protein. The products may be dyed with acid dyes in the normal manner. C.O.C.

Polyacrylonitrile Fibres of Improved Hydrophilic and Dyeing Properties
Stockholm Superfosfat Fabriks *BP* 777,330
Extruding a composition containing polyacrylonitrile (≮ 70% by wt.) and either another polyvinyl compound or an amine, guanidine or amide having an aliphatic chain of 6–30 C into a liquid hydrocarbon coagulant yields products of improved dyeing properties and decreased tendency to become electrostatically charged. C.O.C.

Polyacrylonitrile Fibres
Montecatini Societa Generale per l'Industria Mineraria et Chimica *BP* 778,254
Fibres of improved affinity for disperse and vat dyes are obtained by incorporation of a copolymer of acrylonitrile with butadiene, *iso*prene or a monomer of formula $CH_2{:}CR^1R^2$ (R^1 = H or Alk; R^2 = Alk). C.O.C.

Thermal Stabilisation of Acrylonitrile Polymers
FH *BP* 777,304
The resistance to heat of acrylonitrile polymers containing basic groups is improved by treating them with hydrofluoboric acid or its water soluble salts preferably at 85–95°C. C.O.C.

Linear Thermoplastic Polycarbonates
Bayer *BP* 772,627
Polycarbonates made from (mono-hydroxy phenyl)-substituted aliphatic hydrocarbons have high m.p. and are useful for the manufacture of plastics and fibres. W.G.C.

Bulked Yarn
Algemene Kunstzijde Unie N.V. *BP* 770,018
Improved apparatus for the production of bulked yarn from continuous filament by means of an air-jet is described. W.G.C.

Bulked Yarn
Inventa *BP* 775,075
In the production of crimped yarn from synthetic fibres, twist is set in the yarn by means of wet steam. W.G.C.

Bulked Yarns
Zwirnerei & Naehfadenfabrik Goeggingen *BP* 773,816
Apparatus is described for the production of bulked stretch yarns from thermoplastic yarns by means of a false twisting device. W.G.C.

Hydrolysis of Cellulose and its Relation to Structure (XI p. 441)
Treatment of Cellulose Esters with Polyepoxides (XI p. 442)
Colour Stabilisation of Acrylonitrile Copolymers (XIII p. 442)
Kinetics of Free-radical Reactions in Nylon (XIII p. 442)
Correlation of the Alkali Solubility of Wool and its Resistance to Abrasion (XIV p. 443)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

PATENTS

Washing Raw Wool with Organic Solvents
AB Separator *USP* 2,737,435
The wool passes through a series of vessels through which the scouring liquors flow from one to the other in the opposite direction to the wool. When passing from at least one vessel to another the liquor passes through a purifying device from which part of the liquor is returned to the vessel from which it came and the other part to the vessel preceding it, the latter part of the liquor from the first vessel being passed to the last vessel. This enables the dirty liquor to be discharged from a single point in the system. C.O.C.

Recovery of Solvent in the Continuous Treatment of Textiles with Volatile Solvents
ICI *BP* 773,968
In a solvent recovery chamber the treated textile passes through an aqueous medium at a temperature sufficient to vaporise the residual solvent. There are inlet and outlet vapour seals above the level of the liquid. These seals each comprises a separate compartment containing a quantity of the volatile solvent below the surface of which there is the lower end of a duct through which the textile passes. C.O.C.

Damage caused by Retained Chlorine. Review of the Activities of the Committee 1953–1956 (XIV p. 443)

VIII— DYEING

Reactive Dyes for Textiles

T. Vickerstaff

J.S.D.C., **73**, 237–247 (June 1957)

Of various methods of achieving wet fastness in dyeings, direct chemical linkage of the dye to the fibre has not hitherto been employed in practice. The Procion dyes are a new class of dyes which will react with cellulose in presence of alkali. The evidence for chemical reaction is reviewed. Being dyes of small molecular weight, their affinity for cellulose is low and diffusion inside cellulose fibres rapid, so that cold dyeing becomes feasible. Also, side-reaction of the dyes with water is minimised at low temperatures. The importance of these properties in determining the preferred dyeing methods is discussed. Procion dyes can be applied in practice by continuous processes, including a method which entails only padding and drying. Novel batch dyeing methods have been developed. The dyes can also be applied by printing with a suitable thickener, and again a novel non-steaming printing style has been evolved and a particularly interesting style using white and vat-dye-illuminated resists under Procion dyes. The fastness properties characteristic of chemical linkage are briefly reviewed. AUTHOR

Dyeing of Cellulose Acetate with Disperse Dyes. VI— Affinities, Heats of Dyeing, and Heats of Solution

C. L. Bird and P. Harris

J.S.D.C., **73**, 199–202 (May 1957)

The partition of disperse dyes between water and cellulose acetate has been studied by means of desorption experiments, and the results have been used to calculate values for affinity, heat of dyeing, and entropy change. Aqueous solubilities have been determined at different temperatures in order to obtain heats of aqueous solution, and values for fibre saturation and heat of solution in the fibre have been calculated. AUTHORS

Dyeing of Dicel and Tricel Yarns and Fabrics

H. C. Olpin and J. Wood

J.S.D.C., **73**, 247–258 (June 1957)

Full colours are obtainable on Dicel and Tricel with disperse, azoic, and developed dyes. A carrier technique, no longer considered necessary for Dicel, is valuable for Tricel, for which high-temperature dyeing also can be used. Azoic combinations, applicable to both fibres by the concurrent technique, show variable resistance to permanent change of colour on ironing. Some combinations are substantially free from this trouble, but in all cases where it takes place it is associated with change in the degree of aggregation, which suggests redispersion of the dye aggregates in the fibre. Developed navy blues and blacks are more satisfactory than direct dyeings, and 3-hydroxy-2-naphthoic acid can be used as a developer for both navy blues and blacks on Tricel owing to its greater resistance to change in colour with alkali on that fibre. Disperse dyes of the C.I. Disperse Blue 27 type are acidic, and their alkali-metal salts are very vulnerable to destruction by light and by oxidising agents, which imposes restrictions on their usefulness on Dicel. On Tricel, however, where salt formation does not take place, these blues are quite safe for all purposes. AUTHORS

Fibro and Fibrolane in the Carpet Trade with particular reference to Dyeing

R. C. Cheetham

J.S.D.C., **73**, 189–199 (May 1957)

The impact of man-made fibres on the carpet trade is discussed, and the desirable attributes of a carpet fibre are suggested. The properties and the performance of viscose rayon staple (Fibro) and regenerated protein staple (Fibrolane) are then considered in detail, some indication being given of the relative advantages of these fibres as well as any apparent disadvantages. The methods being developed to overcome these disadvantages include modifications to fibre structure, as in crimped Fibro and Fibre M 55, and also treatments designed to reduce rate of soiling. Suggestions are then made regarding the composition of man-made fibre blends and reference is made to spun-dyed staple. The scouring and the dyeing of blends consisting of Fibro and/or Fibrolane are then considered, detailed methods and selections of dyes being given for the various staples and yarns. Particular reference is made to the effect of denier on dyeing rate as it affects both staple and yarn. The absorption of direct dyes by protein blends containing Fibro is shown to follow similar lines to that by Fibro itself, but certain special considerations apply. Finally, brief reference to the dyeing of tufted carpets is followed by a summary of progress. AUTHOR

Dyes for the Hydrophobic Fibres

H. E. Schroeder and S. N. Boyd

Text. Research J., **27**, 275–285 (April 1957)

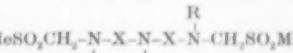
The classes of dyes which show affinity for hydrophobic fibres, and the mechanisms involved, are discussed. For disperse dyes, the important characteristic is the balance between hydrophobic and hydrophilic character of the molecule. Affinity is promoted by a relatively high ratio of mass to polar groups in both disperse and ionic dyes. Fastness to washing, sublimation and dry-cleaning are all favoured by an increase in molecular size and a relative decrease in solubility in water or solvent. Although light fastness presents a more complex problem, it appears that amino groups in the dye molecule have an increasingly deleterious effect as they become more basic. Improvement can be effected by acylation, arylation, or introduction of an electron-attracting group in an *ortho* position. Since the photochemical decomposition of a dye molecule is a reaction of the electronically excited molecule with the fibre, light fastness is a characteristic of the particular dye–fibre system. J.C.F.

PATENTS

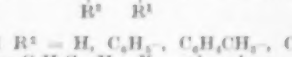
Oxidation Dyes on the Fibre

Acna *BP* 776,459

N-methane sulphonic and sulphinic acids, e.g.—



or



(R, R^1 and R^2 = H, C_6H_5–, $C_6H_4CH_3$–, $C_6H_4OCH_3$–, $C_6H_4C_2H_5$– or $C_6H_4C_{10}H_7$; X = phenylene, naphthylene or anthraquinonyl residue; Me = alkali metal) are used for producing oxidation dyes on the fibre. These compounds are applied, by printing or foulard treatment, mixed with an oxidising agent (chromate, bichromate, chlorate or peroxide) and a catalyst (Cu, Va or Fe salt) and oxidation is carried out under slightly acid conditions as e.g. produced by heating NH_4 oxalate. Advantages over the sulphamic acid derivatives of amines of *BP* 498,755 (J.S.D.C., **55**, 278 (1939)) are greater ease of preparation, shorter steaming time and, in certain cases, greater fastness to soaping. The black dyeings or prints obtained do not turn green. E.T.

Dyeing with Dye-Metal Complexes

BASF *BP* 774,035

When dyeing wool with dyes containing a metal in complex union but no sulphonic or carboxylic acid group presence of an anionic surface active compound and a reaction product of > 50 mol. ethylene or propylene oxide and 1 mol. of a hydroxyalkylatable compound of > 5 C, e.g. oleyl alcohol, oleic acid or stearylamide, ensures that even deep dyeings are fast to rubbing. C.O.C.

Dyeing of Polyester Fibres with Disperse Dyes

ICI *BP* 777,068

Yellow dyeings or prints of excellent light fastness and improved fastness to sublimation are produced on polyester fibres, e.g. terylene from aqueous dispersions of—

(R^1 = H, subst. or unsubst. Alk, *cyclo*alkyl, aralkyl or Ar; R^2 = H, subst. or unsubst. Alk or *cyclo*alkyl; Y = –CO– or $–SO_2–$; X = radical containing basic N joined by a single valency bond to an aromatic ring). The dyes may be produced e.g. by condensing a 4-halogeno-3-nitrobenzamide with a primary or secondary amine *or* by treating a sulphonyl chloride with ammonia or an appropriate amine. E.T.

Production and/or Stabilisation of Hydroquinones (IV p. 434)

Molecular Mixtures of 1:5- and/or 1:4-Diacylaminoanthraquinone Vat Dyes (IV p. 434)
Fluorescent Pyrazoline Dyes for Use in Dyeing and in Coatings for Fibrous Materials (IV p. 435)
Cuprous Thiocyanate for Aftertreatment of Cellulose dyed or printed with Metallisable Direct Dyes (IX below)

IX—PRINTING

PATENTS

Cuprous Thiocyanate for Aftertreatment of Cellulose dyed or printed with Metallisable Direct Dyes

Ciba *BP* 775,034

Cuprous thiocyanate may be used for the aftertreatment of cellulose dyed with metallisable direct dyes, and is particularly valuable in printing, where it can be incorporated in the printing paste with the dye. On steaming, fixation of dye and formation of copper complex take place, to give prints of good wet fastness. E.S.

Aqueous Pigment-Resin Printing Compositions

L. Auer *BP* 774,078

A pigment-resin concentrate suitable for use in printing textiles consists of a pasty aqueous emulsion containing a resinous ester of a polyunsaturated fatty acid, e.g. a linseed oil acid ester, a polyhydric alkoxy derivative of a polynuclear polyhydric phenol, e.g. the condensate of epichlorohydrin with *pp'*-dihydroxydiphenyldimethyl methane, and a volatile organic solvent. The ester should be thermosetting at > 105°c. There should also be present 3–15% by wt. (on the dispersion) of a non-white pigment. The total non-volatile content of the dispersion should be 15–40% of which less than half should be pigment. C.O.C.

Oil-in-Water Emulsion Type Textile Printing Pastes

L. Auer *BP* 774,080

A paste which can be used either as an extender emulsion in pigment printing or as a carrier in printing with dyes comprises water (47·5–61·5), a water-soluble cellulose ether of viscosity as 2% aq. soln. at 20°c. > 1200 cps. (0·3–0·8) and a volatile water-immiscible organic solvent (38–51·5). Use of such a paste gives much better colour value than the oil-in-water emulsion hitherto used. C.O.C.

Colour Photography

Ciba *BP* 774,534

A photographic material contains at least two metallic silver images, at different depths in a single layer. At least one of the images is not close to a reducible dye or dye component, e.g. a vat or azo dye, or diazo or aromatic nitro compound, while at least another image is close to such a dye or dye component. Colour is developed by treating the material by a method known to destroy a silver image by using a reducible dye or dye component as the oxidising agent. This destroys only that silver image close to the dye or dye component and replaces it by a dye image. The other silver image is unaffected by this treatment; it is then in its turn converted to a dye image by any suitable process. C.O.C.

Printing, Coating or Bonding Thermoplastic Vinyl, Vinylidene and Acrylic Resins

Compra Plastics *BP* 774,891

Modification of *BP* 669,738. An ink or cement based on a solution of partly or completely polymerised polystyrene, poly(vinyl chloride), chlorinated poly(vinyl chloride), halogen-substituted styrene polymers, or styrene-unsaturated polyester copolymers of softening point close to that of the material to be printed or bonded is used. C.O.C.

Embossing and Relief Printing Finely Surface-embossed Material

Chambon *BP* 778,382

Foil of aluminium, its alloys or the like which has been finely embossed to give it a silken appearance is passed between two rollers the first of which bears in relief at least part of the design to be relieved on the foil so that the fine embossing is flattened out in those areas. It then passes between this first roller and a third roller which allows the flattened areas to be raised in relief by the first roller. Finally it passes through an ink applying unit which applies a coating of ink to the raised areas. C.O.C.

Colour Photography—Use of Rhodium Salts

ICI *BP* 775,197

When gelatino-silver halide emulsions are precipitated in presence of a rhodium salt and the emulsions so formed are applied to a base, exposed and developed with a colour forming developer in presence of a colour coupler, the contrast is considerably higher than that obtained with an emulsion precipitated in absence of a rhodium salt. C.O.C.

Colour Photography

ICI *BP* 777,583
BP 777,587

Simultaneously Bleaching and Fixing a Photographic Colour Image

Gevaert Photoprodukten *BP* 777,635

Xerographic Development

Battelle Development Corpn. *BP* 774,433

Finely divided powder is deposited on several carrier members each of which is held so that it can flexibly or yieldingly contact the image-bearing surface and present the powder to it. C.O.C.

Pressing Adhesive Foil or Transfers onto a Substrate

A. J. Eklund *BP* 778,295

A press has two co-operating surfaces between which the object and the transfer are present. At least one and preferably both of these surfaces comprises an elastic membrane. Means are provided to prevent air bubbles forming between the object and the transfer. C.O.C.

Colour Couplers (IV p. 432)
Diazoamino Derivatives for use in Printing (IV p. 432)
Fluorescent Pyrazoline Dyes for Use in Dyeing and in Coatings for Fibrous Materials (IV p. 435)
Mixture of a Cyanine and a Benzimidazolocarbocyanine Dye as a Supersensitiser (IV p. 437)
Aqueous Dispersions of Pigments (IV p. 437)
Reactive Dyes for Textiles (VIII p. 439)
Coloured Patterns on Thermoplastic Films (XIII p. 442)

X—SIZING AND FINISHING

Carbamoylethylation of Cotton with Acrylamide

J. W. Frick, W. A. Reeves, and J. D. Guthrie

Text. Research J., **27**, 294–299 (April 1957)

Carbamoylethylation of cotton fabric was accomplished by impregnating the fabric with an aqueous solution containing 50% acrylamide and 4% NaOH, and heating for 4 min. at 135°c. The treated fabric was stable under standard laundering conditions. On boiling in 5% NaOH 70% of the ether groups were cleaved and the remainder were hydrolysed to carboxyethyl groups. The treated fabric showed good retention of strip breaking strength, slight rot resistance, and substantial heat resistance. It could be dyed with a direct dye and with vat dyes without hydrolysis of the carbamoylethyl groups. When the carbamoylethylated fabric was moistened with sodium hypochlorite solution and dried, the carbamoylethyl groups were partially converted to aminoethyl groups, and the fabric could then be dyed with Kiton Fast Red 3GLL. J.C.F.

Soil-resistant Finish for Cotton Fabrics

V. W. Tripp, R. L. Clayton, and B. R. Porter

Text. Research J., **27**, 340–342 (April 1957)

Cotton is made soil-resistant by treatment with perfluorodecanoic acid (I). The fabric is padded with a dilute solution of I in water or an organic solvent, e.g. 0·2% I in *n*-hexane or 0·5% I in 0·12% aqueous sodium acetate, and dried. These finishes are removed by water. A combined water-repellent and soil resistant finish is obtained by first treating the fabric with a dilute aqueous solution of aluminium acetate or zirconium nitrate followed by drying, and then treating with a solution of I in an organic solvent. Electron micrographs show that treatment with I does not alter the surface appearance of the cotton: its effect is probably due to the production of a low-energy surface on the fibre. J.C.F.

Flameproofing of Nylon

D. O. Douglas

J.S.D.C., **73**, 258–263 (June 1957)

Thiourea–formaldehyde resins can be used to give a non-inflammable finish both on nylon net and on nylon

dyed with chrome dyes. The mechanism of the flameproofing of nylon is discussed. The effects of flameproofing agents and of chrome dyes on the melting point, the viscosity, and the ignition temperature of nylon are examined. In the case of nylon dyed with chrome dyes it is suggested that effective flameproofing agents counter the fall in ignition temperature by lowering the melting point. In the case of resin-treated nylon it is probable that the lowering of the viscosity in the presence of flameproofing agents assists the dropping away of the melt.

AUTHOR

PATENTS

Treating Textiles with Aluminium Oxide Aerogels
Deutsche Gold- und Silber-Scheideanstalt *BP* 776,398

The appearance, handle, and resistance to abrasion of textiles is improved by treatment with an aqueous dispersion of aluminium oxide aerogels. W.G.C.

Imparting Dimensional Stability and Resistance to Solvents and to Ageing to Textiles containing Wool
G. W. Pierce *USP* 2,737,434

The goods, preferably previously wet out with water, are impregnated with an aqueous dispersion of a polymeric substance in absence of any influence or substance tending to fix the polymer on the fibres. The polymer is then fixed on the fibre by any suitable means, e.g. by acid treatment or drying. This yields a finish which has good fastness to washing but not to organic solvents. If the polymer used is a sulphur-vulcanisable unsaturated polymer then further treatment with a cystine linkage splitting agency, e.g. radiation by ultraviolet radiation, imparts increased resistance to organic solvents and ageing, and a better handle. C.O.C.

Rendering Fabrics Water-repellent
Dow Corning Corpn. *USP* 2,588,366

Fabrics of all types of organic fibres are rendered water-repellent by wetting them with a mixture of three organosiloxane polymers and then heating at 100–475° F. for 1 hr.–5 sec. Use of a mixture of (1) a methyl hydrogen polysiloxane fluid containing 1·0–1·5 methyl radicals and 0·75–1·25 H atoms bonded to Si per Si atom there being a total of 2·0–2·25 CH_3 radicals and H atoms per Si atom, (2) a methyl polysiloxane fluid containing 2·0–2·1 CH_3 per Si atom and of viscosity 1000 cs., and (3) a resin of formula—

$$(CH_3)_x\ SiO_{\frac{4-x}{2}}$$

(x = 1·0–1·25), gives a very efficient water-repellent finish to nylon and regenerated cellulose without imparting a slippery handle and on cotton a water-repellent finish which is faster to washing than has hitherto been possible and which also has a crisp handle. C.O.C.

Rendering Textiles Water-repellent
Deering Milliken Research Corpn. *USP* 2,750,305

The goods are treated with an ammoniacal aqueous emulsion of a silicone resin (containing an ammonium salt of an organic acid as the emulsifying agent), and, as catalyst, stannous, ferrous or cuprous hydroxide, dried and baked. Any silicone resin containing an active hydride radical may be used. C.O.C.

Gas-resistant Fabric
Trelleborgs Gummifabriks *BP* 773,893

Woven nylon cloth is coated on one or both sides with butyl rubber containing a filler in the form of flakes, e.g. C.I. Pigment White 20, C.I. Pigment Black 10 or C.I. Pigment Metal 1, and then vulcanised. This yields a strong, light, supple and soft fabric used for making into protective clothing, e.g. it is resistant to mustard gas in both its liquid and vapour forms. C.O.C.

Dirt-repelling, Soft, and Elastic Artificial Leather-coated Fabric
Deutsche Gold- und Silber-Scheideanstalt vormals Roessler *BP* 775,597

A temporary support, e.g. paper or polished metal foil, which may be embossed, is thinly coated with a lacquer which adheres more readily to the resinous polymer film to be fused on it than to the support rod which can act as a barrier to migration of plasticiser. One or more layers of a resinous paste of one of the types used in making artificial leather is then applied and converted into a film by heat. A textile backing, preceded if necessary by an adhesive, is then applied and the assembly stripped from the support. The process is particularly applicable to the coating of knitted or woven net fabric. C.O.C.

Stiffening Felt Hat Bodies
Monsanto Chemical Co. *USP* 2,737,462

The goods while at pH < 4 are treated in a weakly alkaline aqueous solution or dispersion of a copolymer of 85–99 mol. % of vinylacetate and 15–1 mol. % of an ethylenically unsatd. aliphatic mono- or di-carboxylic acid, e.g. crotonic acid, or an anhydride or half ester of such an acid, e.g. butyl acid maleate, until the liquor squeezed from the goods is clear but not for more than 120 sec. The goods are then removed from the liquor, gently squeezed to a pickup of 100–175% on their dry weight. The copolymer is then set or precipitated in the goods which are then hydroextracted and finished as usual. This gives a finish having all the advantages and none of the disadvantages, e.g. yellowness, of a shellac finish. C.O.C.

Coating Composition (III p. 430)
Alginate Hessian (VI p. 438)
Treating Leather, Textiles and other Fibrous Materials Dicyandiamide–Formaldehyde Resins (XII p. 442)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Hydrolysis of Cellulose and its Relation to Structure
A. Sharples

Trans. Faraday Soc., **53**, 1003–1013 (July 1957)

Hydrolytic studies on cellobiose, cotton, and a cotton hydrocellulose indicate that the reactive bonds in cotton are confined to amorphous regions. Glucosidic bonds in surfaces of crystallites are resistant to degradation. After removal of amorphous material, degradation occurs by attack on the ends of crystallites at a rate which, to a first approximation, is inversely proportional to their mean length. This and other features of the reaction can be accounted for if it is assumed that the crystallite lengths are exponentially distributed. W.R.M.

Transitions in Cellulose Acetates
J. Russell and R. G. van Kerpel

J. Polymer Sci., **25**, 77–96 (June 1957)

The temperature dependence of specific volume, rigidity modulus, and mechanical loss of plasticised and unplasticised cellulose acetates has been studied. Measurements of specific volume indicated three apparent second-order transitions and one first-order transition in cellulose triacetate, and three or possibly four apparent second-order transitions but no first-order transition in a secondary acetate. First- and second-order transitions were depressed by addition of plasticiser. The existence of the second-order transitions was confirmed by measurements of rigidity modulus and mechanical loss, which show that only the highest-temperature transition has the characteristics of a normal glass–rubber transition. W.R.M.

PATENTS

Starch Sized Paper
S. D. Warren Co. *BP* 773,732

Paper with increased strength, stiffness and smoothness is obtained by adding a colloidal dispersion of mannogalactum, heated to below the gelatinisation point of starch, and mixed with finely divided ungelatinised starch, to the fibrous paper making furnish. The web, after formation, is heated to gelatinise the starch. The finished paper may contain 1–50% of starch. G.S.B.

Coating Paper
A/S Protan *BP* 776,799

An improved surface for gloss printing is obtained by the use of grease resistant colloid materials or plastics. The process and materials avoids soiling of paper machine cylinders and felts. The coating composition consists of an alginate and a vinyl resin latex in aqueous dispersion, and is applied at the machine drier section. An example of the composition (parts by weight) is alginate (1–2), polyvinyl acetate emulsion (3–10), softening agent, e.g. glycerin (0·5–1·0), TiO_2 (1–100), and water (50–1000). G.S.B.

Paper Coating
Mead Corpn. *BP* 777,613

A starch-clay coated paper with improved printing characteristics is obtained by use of a smoothing roll,

maintained at a low temperature to stabilise the coating against adhesion. In addition a thin uniform film of release fluid such as ethylene glycol is metered to the smoothing roll before contact between the coated surface and the smoothing roll. G.S.B.

Finishing Coated Paper
S. D. Warren Co. *BP* 774,352

High gloss and good level is achieved by application of aqueous liquid to the dry surface of a coated paper, whose coating includes a hydrophilic, swellable adhesive, e.g. casein. The web, after moistening, is passed through a pool of aqueous liquid, which is followed immediately by pressing against a wet, heated, highly polished finishing cylinder. G.S.B.

Treatment of Cellulose Esters with Polyepoxides
N.V. de Bataafsche Petroleum Maatschappij *BP* 770,014

Solvent and heat resistant products are obtained by incorporating in solutions of cellulose esters containing free hydroxyl group a glycidyl polyether of a polyhydric alcohol or polyhydric phenol having at least 1·1 epoxy groups per molecule, and an epoxy-curing catalyst. After removal of the solvent, the resulting films or fibres are baked to complete the reaction. W.G.C.

Stabilisers for Acrylonitrile Polymers (XIII this page)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

PATENTS

Dehairing
Röhm & Haas *BP* 776,323

Dehairing is readily carried out by applying a powdered protease composition to the green or soaked hides, preferably to the flesh side, and then after a suitable interval removing the hair. This method gives satisfactory results on calfskins, fresh unpreserved sheepskins and dried and salted sheepskins. C.O.C.

Tanning Leather with Zinc Sulphides
American Zinc, Lead & Smelting Co. *BP* 778,153

White leather of resistance to boiling water between that of vegetable tanned and chrome leather is produced by treating a hide with a soluble sulphide or hydrosulphide and then with a Zn salt. C.O.C.

Tanning
Boehme Fettchemie *BP* 777,699

The raw skins or hides are treated first with a nitrogenous resin precondensate, e.g. a melamine–formaldehyde precondensate, and then with a surface-active compound containing an aliphatic, *cyclo*aliphatic or araliphatic radical of > 5 C, e.g. a sodium alkylsulphate, the precondensate being cured either during or after these processes. The resulting leathers have high flexibility, excellent tear resistance and smooth close grain. They are white or pale coloured, dye well and are stable to washing. C.O.C.

Retanning of Mineral Pre-tanned Leather
Koppers Co. *BP* 776,956

If mineral pretanned leather is retanned with resorcinol and formaldehyde at pH 6·0–7·5 very strong solutions can be used. Thus the leather is wet out, drained, mixed with 2–5% of its wet weight of solid resorcinol and then treated with 1·5–3·0 mol. HCHO per mol. resorcinol while maintaining the pH of the liquor at 6·0–7·5. C.O.C.

Treating Leather, Textiles, and other Fibrous Materials with Dicyandiamide-Formaldehyde Resins
Jacques Wolf & Co. *USP* 2,737,464

Modification of *USP* 2,567,238. Leather or other fibrous material in a state in which it will be reactive with or neutralise cationic material is treated with the resin obtained from dicyandiamide, formaldehyde and boric acid. Leather so treated is very full, tight and smooth. The process may be used to render cotton and rayon dimensionally stable and to give them affinity for anionic dyes. C.O.C.

Water-repellent, Washable, Flexible Leather
General Electric Co. *BP* 777,078

The leather is treated with an aqueous emulsion consisting primarily of (*a*) a mixture of 50–75% dimethyldichlorosilane, 1–10% trimethylchlorosilane, 10–35% methyltrichlorsilane and up to 10% of a methylchlorodisilane, and (*b*) a metal salt of an organic or inorganic acid as the curing agent. C.O.C.

Leather Dressing
FBy *BP* 777,293

A leather dressing compatible with aqueous leather body colours consists of an aqueous emulsion of copolymers made from $\leqslant 45\%$ of butadiene or a homologue or derivative thereof and $\leqslant 10\%$ of 1:1-dichloroethene and, if desired, additional vinyl compounds. C.O.C.

Dirt-repelling, Soft, and Elastic Artificial Leather-coated Fabric (X p. 441)
Action of Perspiration on Vegetable-tanned Leathers (XIV p. 444)

XIII— RUBBER; RESINS; PLASTICS

Propagation Rate in Acrylonitrile Polymerisation
W. M. Thomas and R. L. Webb
J.Polymer Sci., **25**, 118–119 (June 1957)

An estimate is made of k_p, the rate constant for propagation, and a value for use in aqueous systems at room temperature is given. W.R.M.

Kinetics of Free-radical Reactions in Nylon
R. A. Ford
J. Colloid Sci., **12**, 271–282 (June 1957)

The formation of an absorption centre at 2900 A. in nylon films by action of ultraviolet radiation is reported. The processes involved are found to be of a free-radical nature, and it is suggested that the centre is either an alkyl or acyl peroxy radical. The kinetics of absorption centre formation have been studied by a spectrophotometric technique. W.R.M.

PATENTS

Sequestering Agents for Stabilising Rubber to Water containing Copper or Other Heavy Metal
Gy *BP* 775,361

Incorporation in natural rubber mixes of 0·1–2·0% by weight of an alkali metal or alkaline earth metal complex of a polyamine polycarboxylic acid which forms stable watersoluble complexes with heavy metals, e.g. nitrilotriacetic or ethylenediaminetetraacetic acid, stabilise articles made from the mixes when they are exposed to water containing Cu, Fe, Co or Mn. C.O.C.

Iron Phthalocyanine as Peptising Agent for Rubber
DuP *BP* 776,101

Iron phthalocyanine, iron trinitrophthalocyanine (from 4-nitrophthalimide and phthalic anhydride in a urea melt), iron mono 4-chlorophthalocyanine, and their leuco derivatives are excellent peptising agents for rubber. C.O.C.

Vulcanising Agent
Deutsche Gold- und Silber-Scheideanstalt vormals Roessler *BP* 777,308

The condensate of a polyamine and a ketone, e.g. of *p*-phenylenediamine and methylketone, is an excellent vulcanising agent for natural and synthetic rubbers, Neoprene, etc. C.O.C.

Stabilisers for Acrylonitrile Polymers
Dow Chemical Co. *BP* 774,768

Incorporation of 5–15% by weight of a compound of formula $R^1R^2N{\cdot}CH_2{\cdot}CH_2{\cdot}CN$ (R^1 = Alk of 1–4 C; R^2 = H or R^1) e.g. β-dimethylamino propionitrile, stabilises acrylonitrile polymers to heat and light. *BP* 774,769

Compounds of formula $X^1X^2N{\cdot}CY^1{\cdot}Y^2Z$ (X^1 and X^2 = same or different H, CH_3, C_2H_5, 2-hydroxyethyl, propyl and *iso*propyl, the sum of C atoms in $X^1 + X^2 = 1$–4; Y^1 = O or S; Y^2 = S or may = O when Y^1 = S; Z = Alk of 1–4 C; total number of C atoms is < 8), e.g. *iso*propyl-*N*-methylthionocarbamate, are similarly used. C.O.C.

Colour Stabilisation of Acrylonitrile Copolymers
Dow Chemical Co. *BP* 776,915

Oxalic, glyoxylic or formic acid prevent discoloration when acrylonitrile copolymers are formed by heating at > 200°C. C.O.C.

Coloured Patterns on Thermoplastic Films
H. W. Brock *BP* 774,527

The printed pattern is applied to a backing member and then a hot film of thermoplastic material extruded onto it so that the printed pattern becomes embodied in the film. The method is particularly applicable to films of plasticised polyvinyl chloride. C.O.C.

Coating Plastic Material
H. W. Brock *BP* 775,371

Hot plastic material is extruded and while still hot is treated with flock, metallic powder or other powdered material. Suitable apparatus is described. C.O.C.

Grained Linoleum
Armstrong Cork Co. *BP* 777,748

Individual blocks of precalendered variegated colour composition are fed uniformly across the nip of a sheeting calender. The feed is controlled so that only sufficient blocks fill the nip so as to form a sheet in which the individual colour composition blocks can be distinguished on its surface. C.O.C.

Plastic Sheet Material containing Pearl Essence
Patrician Plastic Corpn. *BP* 780,458

A mixture of a transparent unsaturated polyester resin Pearl Essence (C.I. Natural White 1) and a peroxide catalyst is run into a hot horizontally mounted rotating drum. The viscosity of the mix, the temperature, and the speed of the drum are such that a non-flowable polymerised cylindrical layer of the pearly material is formed on the inside of the drum. This material is removed from the drum while still flaccid. C.O.C.

Coating Composition (III p. 430)

Filaments, Fibres, etc. from Acrylonitrile Polymers (VI p. 438)

Polyacrylonitrile Films or Filaments of Improved Dyeing Properties (VI p. 438)

Linear Thermoplastic Polycarbonates (VI p. 438)

Printing, Coating or Bonding Thermoplastic Vinyl, Vinylidene and Acrylic Resins (IX p. 440)

XIV— ANALYSIS; TESTING; APPARATUS

Standardisation of Test Methods for Textile Auxiliaries. II— Determination of Vat Acid Dispersion Power of Surface-active Agents
G. Schwen and C. Rackemann

Melliand Textilber., **37**, 1345–1347 (Nov. 1956): *Amer. Dyestuff Rep.*, **46**, 278–280 (22 April 1957)

Vat dyes (insoluble pigments) can be converted to sodium leuco vat dyes (soluble) by alkali and a reducing agent. The vat dyes are made insoluble by the addition of acid to give disperse dyes. The dispersion can be prevented from precipitating by the addition of surface-active agents to the colloidal suspension. Three dyes (C.I. Vat Green 1, C.I. Vat Green 3, and C.I. Vat Blue 20) were used to test 19 surface-active agents. The minimum concentration of surface-active agent necessary to prevent precipitation of the dispersion was measured. The results showed a different order of efficiency with different dyes. For all three dyes the most effective stabilising agent was the disodium salt of di(2-sulpho-1-naphthyl)-methane. The results were reproducible. P.T.S.

Stilbenebisazo-β-naphthol as Indicator in the Volumetric Determination of Sulphate Ions
L. V. Markova

Ukrainian Chem. J., **23**, 89–91 (Jan. 1957)

Stilbene - 4:4′ - bis - 1 - azo - 2 - hydroxynaphthalene - 2:2′-disulphonic acid is used as an indicator in the microtitration of sulphate ions with $BaCl_2$ solution in presence of acetone or *iso*amyl alcohol, the equivalence point being shown by the decoloration of the alcohol (or acetone) layer. G.J.K.

Correlation of the Alkali Solubility of Wool and its Resistance to Abrasion
K. Lees and F. F. Elsworth

Proc. International Wool Textile Research Conf. Australia, **E**, E 425–E 430, E 600–E 601 (1955)

For a given chemical treatment, there is a correlation between the alkali solubility of the wool and its resistance to abrasion. Different treatments, however, giving rise to the same alkali solubility, may have widely different effects on the resistance to abrasion. A new method of determining abrasion resistance is described. P.G.M.

Damage caused by Retained Chlorine. Review of the Activities of the Committee 1953-1956
L. L. Walmsley

Amer. Dyestuff Rep., **46**, P 237–P 243 (8 April 1957)

Chlorine retained after outside and home laundering damages cloth and lowers its resistance to abrasion. It has proved difficult to correlate results found in the laboratory for damage following chlorine retention with the performance of fabrics in use. Results also vary widely between laboratories. Suggestions for future investigations are made— effects of time, temperature, concentration, etc.; effect of differences and additives in rinsing water; effects of different resins and catalysts used in resin finished cloths; effect of different dyes. P.T.S.

Differentiation of Inks by Electrochromatophoresis
C. L. Brown and P. L. Kirk

Mikrochim. Acta, 1729–1734 (1956): *Chem. Abs.*, **51**, 7033 (10 May 1957)

On testing 23 inks by the electrochromatophoretic method each gave a unique pattern characterised by greater separation and more detail than are possible with other methods. C.O.C.

Micromethod for the Estimation of Cellulose
G. G. Dearing

Nature, **179**, 579 (16 March 1957)

Samples of purified bacterial cellulose, weighted with talc, are suspended in water, the max. concn. of cellulose being 150 μg./ml. To this suspension is added 3 vol. of 96% (wt./vol.) H_2SO_4, and the whole is heated for 6·5 min. on a boiling water-bath and cooled. The resulting pink soln. is centrifuged, and the optical density of the supernatant liquid measured at 520 mμ. A.J.

Determination of the Insolubility of Chemically Modified Cotton in Cuprammonium Hydroxide Solution
W. T. Schreiber

Text. Research J., **27**, 324–326 (April 1957)

Finely ground samples (0·15 g.) of chemically modified cellulose are mixed with 0·3 g. of filter aid and shaken with cuprammonium hydroxide solution (containing glucose to inhibit decomposition of the cellulose) overnight. After centrifuging and decanting the supernatant liquid, the residue is washed with ammonium hydroxide and centrifuged until free from colour. The residue is transferred to a tared sintered glass crucible, dried and weighed. Blank determinations are run using the same weight of filter aid but omitting the cellulose sample, and the weight of cellulose insoluble in the reagent is obtained by difference. Results are given for cotton modified by treatment with epichlorohydrin in presence of NaOH. J.C.F.

Detection of Cobalt with *o*-Phenylenediamine
R. L. Dutta

J. Indian Chem. Soc., **34**, 151–153 (Feb. 1957)

Determination of the Electric Charge of Auxiliary Agents
G. Otto

Leder, **7**, 145 (1956): *J. Soc. Leather Trades Chem.*, **41**, 268 (July 1957)

The electric charge of auxiliary agents may be determined in a test tube by adding certain (proprietary) synthetic polymer indicators. C.J.W.H.

Chromatography of Polynitronaphthylamines
E. R. Ward and P. R. Wells

J.C.S., 2836–2837 (June 1957)

Larson and Harvey's method (*Chem. and Ind.*, **45** (1954)) for the quant. separation of mixtures of nitroanilines by chromatography has now, with minor modifications, been applied successfully to the separation of mixtures of di- and/or tri-nitronaphthylamines (14 examples). It fails with mixtures of mononitronaphthylamines, although mixtures of related cpd., e.g. 7-nitro-1-naphthylamine and 2:4-dibromo-7-nitro-1-naphthylamine, have also been so separated. H.H.H.

Testing Dyewood Extracts
VGCT Committee on Leather Dyeing

Leder, **7**, 234 (1956): *J. Soc. Leather Trades Chem.*, **41**, 267 (July 1957)

A proposed method is put forward. C.J.W.H.

Analysis of Hair Tints
I. Jacobelli-Turi
Rendiconti Ist. Super. Sanità, Roma, **19**, 461–469 (1956): *J. Amer. Leather Chem. Assocn.*, **52**, 347 (June 1957)

The proposed methods may be used not only for hair tints but also for research in the field of fur dyes. The dyes may be detected chromatographically, and in this first report the data are given for several commonly used compounds. The values of R_f were determined by examining the chromatographic spots obtained using as solvent a 4 : 1 : 5 mixture of butanol, acetic acid, and water at 20°c. The R_f values are: *p*-phenylenediamine, 0·38; 2:5-toluylenediamine, 0·48; *p*-aminophenol, 0·55; pyrogallol, 0·71; resorcinol, 0·83. As it may happen that several other compounds have R_f values very close to those indicated above (e.g. pyrocatechol, R_f 0·82), several colour reagents were studied which may be used to identify the single constituents: (*a*) saturated (cold) aqueous solution of vanillin containing HCl (2·5 ml. per 100); (*b*) 0·5 g. of *p*-dimethylaminobenzaldehyde and 30 ml. HCl in 100 ml. H_2O; (*c*) 2 g. phenol in 100 ml. of 5% KOH containing 20 ml. of a dilute solution of Na hypochlorite; (*d*) 1 drop aniline in 250 ml. H_2O acidified with 10 ml. acetic acid and 3 g. Na persulphate; (*e*) 1% solution of silver nitrate. The sensitivity of these reagents permits the detection of the above indicated compounds at concentrations of 0·2–0·5 mg./litre. C.J.W.H.

Characterisation of Cellulose by Rheological Measurements
K. Edelmann
Faserforsch. und Textiltech., **8**, 184–191 (May 1957)

Methods are given by means of which estimates of the average molecular weight and degree of inhomogeneity may be obtained from viscosity measurements on solutions at different rates of shear. W.R.M.

Comparison of the Willows-Markert Swelling Reaction with the Average Degree of Polymerisation of Cotton
G. Stingl and H. Vollenbruck
Textil-Rund., **12**, 388–391 (July 1957)

Diagrams showing the appearance of the swollen mushroom ends of the fibres for different D.P. are given. S.R.C.

Structure and Identification of Wool and other Animal Textile Fibres
A. B. Wildman
Proc. International Wool Textile Research Conf. Australia, **F**, F 156–F 175, F 220–F 222 (1955)

The sampling of fibre mixtures in various forms for the identification of their component fibres is outlined. The preparation of subsamples for microscopic examination is described. The value of various kinds of preparations, including casts and impressions of scale patterns, for fibre identification is discussed and a new classification of medullae and scale patterns is presented. The interpretation of scale patterns for identification purposes is given special attention. The microscopy of different kinds of wool fibres, particularly their scale patterns, is discussed. P.G.M.

Wet Ironing Standard, SNV 98 8 75
Swiss Standards Assocn.
Textil-Rund., **12**, 280 (May 1957)

The shrinkage of wool fabric is covered. S.R.C.

Degree of Crystallinity in Polymers by Nuclear Magnetic Resonance
W. P. Slichter and D. W. McCall
J. Polymer Sci., **25**, 230–234 (July 1957)

A note on the use of nuclear magnetic resonance in the determination of crystallinity. W.R.M.

Nitrogen Estimation in Polyacrylonitrile
U. Bartels
Faserforsch. und Textiltech., **8**, 194–195 (May 1957)

Additions to the sulphuric acid used in the Kjeldahl method of estimating nitrogen in polyacrylonitrile are discussed. A mixture of copper sulphate and potassium nitrate gives the best results. W.R.M.

Degree of Crystallinity of Polychlorotrifluoroethylene by Infrared Method
H. Matsuo
J. Polymer Sci., **25**, 234–237 (July 1957)

Degree of crystallinity is measured by use of the absorption band at 440 cm.$^{-1}$ as the crystalline band and that at 754 cm.$^{-1}$ as the amorphous band. Good correlation between specific volume and degree of crystallinity is obtained. W.R.M.

CH High-temperature Dyeometer and its Use
M. Peter
Textil-Rund., **12**, 305–316 (June 1957)

In general, the exhaustion rate in the orthodox Dyeometer is somewhat faster than in bulk dyeing. The apparatus has been modified to study exhaustion rates above 100°c., examples of which are given. S.R.C.

Determination of the Resistance to Hard Water of Anionic Dyes for Leather
VGCT Committee on Leather Dyeing
Leder, **7**, 233 (1956): *J. Soc. Leather Trades Chem.*, **41**, 267 (July 1957)

A proposed method is put forward. C.J.W.H.

Determination of the Solubility of Anionic Dyes for Leather
VGCT Committee on Leather Dyeing
Das Leder, **7**, 233 (1956): *J. Soc. Leather Trades Chem.*, **41**, 267 (July 1957)

A proposed method is put forward. C.J.W.H.

Action of Perspiration on Vegetable-tanned Leathers
R. Ferebauer
Kozarstvi, **6**, 34–36, 54–55 (1956): *J. Amer. Leather Chem. Assocn.*, **52**, 406 (July 1957)

Action of perspiration on vegetable-tanned insoles was studied. The pH value of fresh human perspiration is 5·2–6·0, but that of perspiration extracted from socks worn for 5 days is 6·6–8·7. The most effective artificial perspiration contained 0·05 N-NH_4OH, 0·4% NaCl, and 0·05% α-aminocaproic acid. Chemical analyses of insoles worn for 4, 8, and 10 months are given. Ash content increased from 1·5 to 3·6% and pH value from 4·86 to 5·80, and tear strength decreased from 2·6 to 1·4 kg./sq.mm. Surface shrinkage occurs as a result of repeated wetting by perspiration or by water at 37°c. and drying. Maximum longitudinal shrinkage after treatment with artificial perspiration for 35 days is 12·4 mm. Prechroming of vegetable-tanned insoles does not influence the deterioration. C.J.W.H.

Provisional Methods of Analysis used at the Experimental Station for the Leather Industries of Naples (Italy)
Cuoio, Pelli, Mat. Concianti, **32**, 89–115, 180–215, 271–296, 402–418 (1956): *J. Amer. Leather Chem. Assocn.*, **52**, 283 (May 1957)

Methods for the determination of several properties of leather are described in detail, including resistance to rubbing, resistance of dyes to water (cold and warm) and to artificial perspiration, and washing test. C.J.W.H.

Colorimetric Methods for the Determination of Formaldehyde fixed by Collagen
S. M. Bose, K. T. Joseph, and B. M. Das
Tanner (India), **11**, No. 3, 23; No. 4, 16 (1956): *J. Soc. Leather Trades Chem.*, **41**, 268 (July 1957)

Six methods were investigated for their suitability for the determination of formaldehyde fixed by collagen. A colorimetric method using Schiff's reagent was suitable when the formaldehyde was not present in very low concentrations. A simple, rapid, and accurate method is described using chromotropic acid. C.J.W.H.

XV— MISCELLANEOUS

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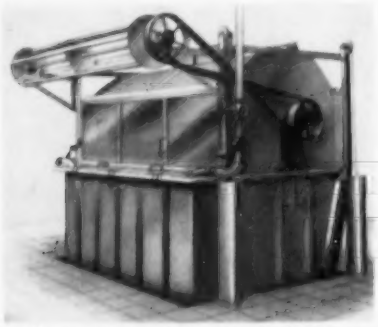
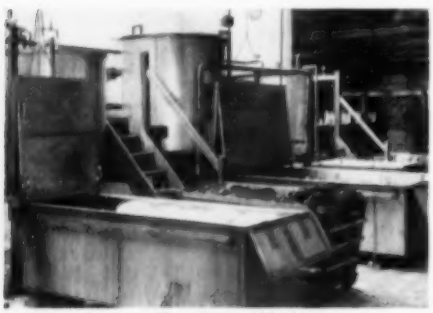


Above — Enclosed Piece Dyeing Machines

Above — Loose Wool Dyeing Plant



Above — Top Dyeing Machines

Hank Dyeing Machines for Carpet and Hand Knitting Yarns

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FORTHCOMING MEETINGS OF THE SOCIETY—*continued from page viii*

Tuesday, 17th December 1957

HUDDERSFIELD SECTION. *The Dyeing of Blends of Fibro with other Man-made Fibres and with Cotton.* W. Lonie, Esq., B.Sc., Ph.D. and R. A. Rutherford, Esq., B.Sc. (British Rayon Research Association). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

Friday, 10th January 1958

LONDON SECTION. *Colour for the Dyer.* Professor W. D. Wright (Imperial College of Science and Technology). (Joint meeting with the London Section of the Oil and Colour Chemists' Association.) Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 14th January 1958

NORTHERN IRELAND SECTION. *The Chemical Foundations of Fast Colour Printing.* A. S. Fern, Esq., B.Sc., D.I.C., F.S.D.C. (Imperial Chemical Industries Ltd.). Further details later.

Thursday, 16th January 1958

MANCHESTER JUNIOR BRANCH. *High Temperature Dyeing on Synthetic Fibres.* H. R. Hadfield, Esq., M.Sc.Tech. Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. *Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Victoria Hotel, **Bradford.** 7.30 p.m.

Friday, 17th January 1958

LONDON SECTION. Dinner and Dance. Waldorf Hotel, **London, W.C.2.**

Tuesday, 21st January 1958

HUDDERSFIELD SECTION. *Tests for Colour Vision.* F Jordinson, Esq, M.Sc., F.R.I.C., F.S.D.C. and T. Minshall, Esq., A.M.C.T., A.T.I. (Dyeing Department, Technical College, Huddersfield). (Joint Meeting with the Huddersfield Section, Royal Institute of Chemistry.) Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

SCOTTISH SECTION. *Flame-resistant Finishes.* J. R. W. Perfect, Esq., B.Sc. (Bradford Dyers' Association Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m. Developments with the use of phosphorus-containing resins.

Wednesday, 22nd January 1958

MIDLANDS SECTION. *Colloquium . . . Auxiliary products—Use and Abuse.* (Short papers by local members.) College of Technology, **Leicester.** 7 p.m.

SCOTTISH JUNIOR BRANCH. *Practical Aspects of Fastness Testing To-day.* P. R. Dawson, Esq. (Imperial Chemical Industries Ltd.). Technical College, **Paisley.** 7.30 p.m.

Friday, 24th January 1958

WEST RIDING SECTION. Ladies' Evening. Victoria Hotel, **Bradford.** 7.30 p.m.

Thursday, 30th January 1958

WEST RIDING SECTION. *A New Approach to Pigment Dyeing of Piece Goods.* Dr. Sulzer (Ciba Ltd., Basle). Victoria Hotel, **Bradford.** 7.30 p.m.

Monday, 3rd February 1958

HUDDERSFIELD SECTION. *Shrink Resistance of Wool.* A. N. Davidson, Esq., B.Sc. (Wool Industries Research Association). (Joint Lecture with the Huddersfield Textile Society.) Technical College, **Huddersfield.** 7.30 p.m.

Tuesday, 4th February 1958

LONDON SECTION. *Progress in Textile. A review of developments and their effect on industry and user—Dyeing and Finishing.* T. Vickerstaff, Esq., M.Sc., Ph.D., F.S.D.C. (Joint Meeting with the London Section, Textile Institute.) Chemical Society, Burlington House, **London, W.1.** 6.30 p.m.

Friday, 7th February 1958

LONDON SECTION. *Spun-dyed Fibres, their Properties and Uses.* C. C. Wilcock, Esq., A.R.T.C.S., F.T.I., F.S.D.C. (Courtaulds Ltd.). Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 11th February 1958

NORTHERN IRELAND SECTION. *A New Approach to Pigment Dyeing.* Dr. G. Sulzer (Ciba Limited). Further details later.

Thursday, 13th February 1958

MANCHESTER JUNIOR BRANCH. *The Scientific Background to Mangling.* E. Moss, Esq. Manchester College of Technology, **Manchester.** 4.30 p.m.

MIDLANDS SECTION. *A New Approach to the Pigment Dyeing of Piece Goods.* Dr. G. Sulzer (Ciba Ltd, Basle). Gas Board Theatre, **Nottingham.** 7 p.m.

WEST RIDING SECTION. *Investigations on the Dyeing Process of Polyester Fibres with Disperse Dyes.* Dr. Glenz (Bayer Co., Leverkusen). Metropole Hotel, King Street, **Leeds.** 7.30 p.m.

Tuesday, 18th February 1958

HUDDERSFIELD SECTION. *The Continuous Dyeing of Wool.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

SCOTTISH SECTION. *A New Approach to Pigment Dyeing of Piece Goods.* Dr. G. Sulzer (Ciba Ltd., Basle). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Friday, 28th February 1958

WEST RIDING SECTION. J. B. Speakman, Esq., D.Sc., F.R.I.C., F.T.I. Details later.

Friday, 7th March 1958

LONDON SECTION. *Recent Developments in the Dyeing of Man-made Fibres.* Dr. R. Wittwer (Ciba Ltd.). Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 11th March 1958

HUDDERSFIELD SECTION. *Recent Developments in the Dyeing of Man-made Fibres.* Dr. R. Wittwer (Ciba Limited, Switzerland). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

NORTHERN IRELAND SECTION. *Some Observations in the Uses of Synthetic Resin Products and Chemical Reactants to Cellulosic Materials.* F. Sloan, Esq., M.Sc. (Kirkpatrick Bros. Ltd.). Further details later.

Thursday, 13th March 1958

MANCHESTER JUNIOR BRANCH. *Terylene.* J. R. Whinfield, Esq. Manchester College of Technology, **Manchester.** 4.30 p.m.

WEST RIDING SECTION. Discussion, *Fluorescent Brightening Agents.*

Panel
- A Member of the British Cotton Industries Research Association.
- J. S. Ingham, Esq. (Marks & Spencer Ltd.).
- Dr. Fearnley (Geigy Co. Ltd., Manchester).
- J. Rayment, Esq. (G. & W. N. Hicking).

Victoria Hotel, **Bradford.** 7.30 p.m.

Tuesday, 18th March 1958

SCOTTISH SECTION. *Easy-care Finishes on Fabrics Containing Courpleta, with Special Reference to Pleating, Embossing and Setting.* A. S. Cluley, Esq., F.T.I. and S. N. Bradshaw, Esq. (Courtaulds Ltd., Coventry). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Wednesday, 19th March 1958

MIDLANDS SECTION. *Optical Brightening Agents.* D. A. W. Adams, Esq., B.Sc., Ph.D. (Joint meeting with The British Association of Chemists.) Midland Hotel, **Derby.** 7 p.m.

Friday, 21st March 1958

MIDLANDS SECTION. Section Annual Dinner. George Hotel, **Nottingham.**

Tuesday, 25th March 1958

HUDDERSFIELD SECTION. Annual General Meeting.

Thursday, 27th March 1958

WEST RIDING SECTION. Annual General Meeting. Victoria Hotel, **Bradford.** 7.30 p.m.

Wednesday, 16th April 1958

MIDLANDS SECTION. *Basic Principles of Dyeing with Disperse Dyes.* D. Finlayson, Esq., M.C., M.A., D.Sc. and H. C. Olpin, Esq., M.Sc., F.R.I.C., F.S.D.C. King's Head Hotel, **Loughborough.** 7 p.m. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section.)

Friday, 18th April 1958

LONDON SECTION. *Colour Fastness Requirements of Luton Textiles.* I. Glasman, Esq., A.T.I. (Marks & Spencer Ltd.). George Hotel, **Luton.** 7 p.m.

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Applications to the Managing Director, Stevensons (Dyers) Limited, Amber Dye Works, Ambergate, Derbyshire.

TECHNICAL SERVICE AND PRODUCT DEVELOPMENT— Chemical Manufacturers near Manchester require Textile Chemist to set up technical service department for textile auxiliaries. Applicants should have University degree or equivalent, with good practical experience in the dyeing and finishing of cellulosic and synthetic fibres. Age 30–40. The position involves laboratory investigations and also visits to customers, and a car will be provided. Salary will be in accordance with qualifications and a Pension Scheme is operated. Write Box V136

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